

## **A Concise Total Synthesis of (–)-Maoecrystal Z**

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### **Supporting Information 2 (Spectral Data):**

jc-1-245-A

Sample Name:

jc-1-245-A

Data Collected on:

indy.caltech.edu-inova500

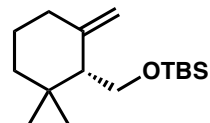
Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-245-A

FidFile: PROTON01



SI-1

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Mar 31 2011

Temp. 25.0 C / 298.1 K

Sample #27, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

32 repetitions

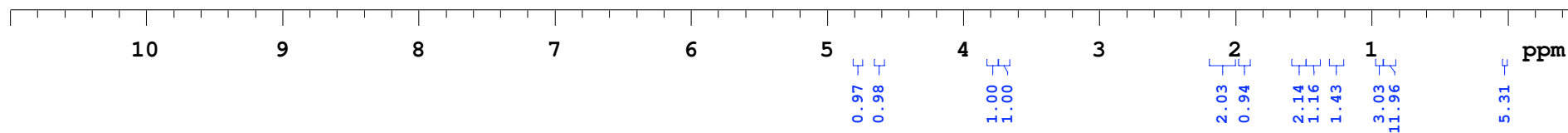
OBSERVE H1, 499.7290203 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min 52 sec



jc-1-245-A

Sample Name:

jc-1-245-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

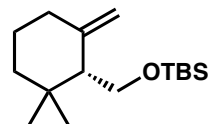
jc-1-245-A

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Mar 31 2011



SI-1

Temp. 25.0 C / 298.1 K

Sample #27, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

256 repetitions

OBSERVE C13, 125.6569604 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

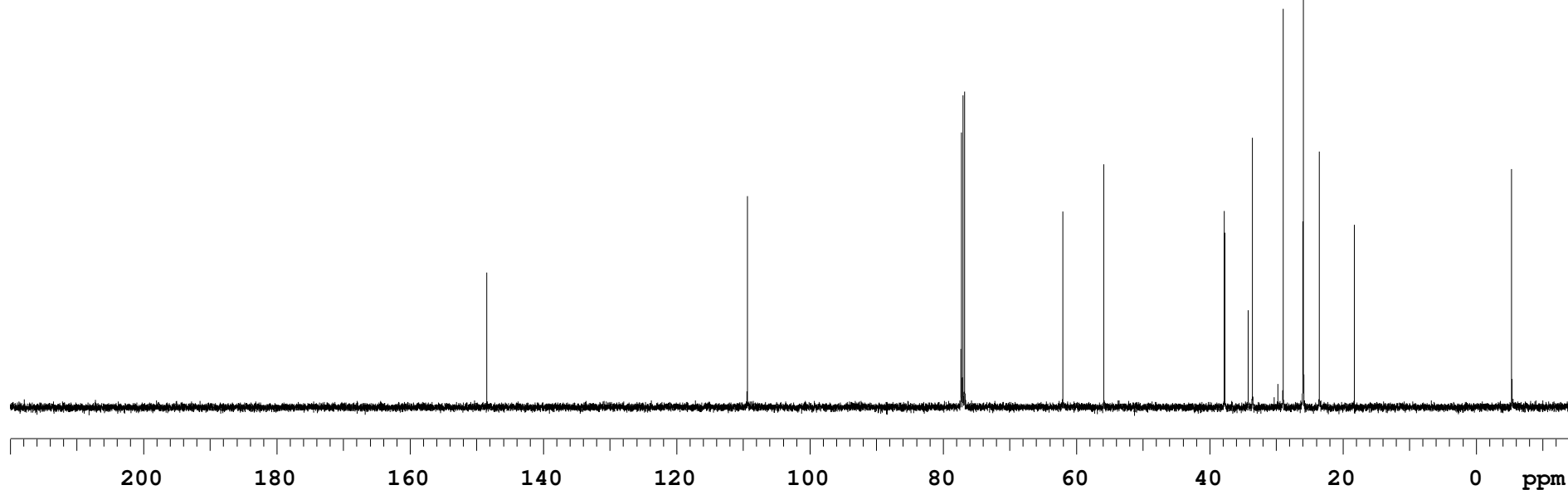
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 8 min 45 sec



jc-1-246-B

Sample Name:

jc-1-246-B

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-246-B

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Apr 1 2011

Temp. 25.0 C / 298.1 K

Sample #46, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

64 repetitions

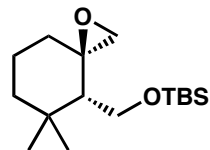
OBSERVE H1, 499.7290203 MHz

DATA PROCESSING

Line broadening 0.2 Hz

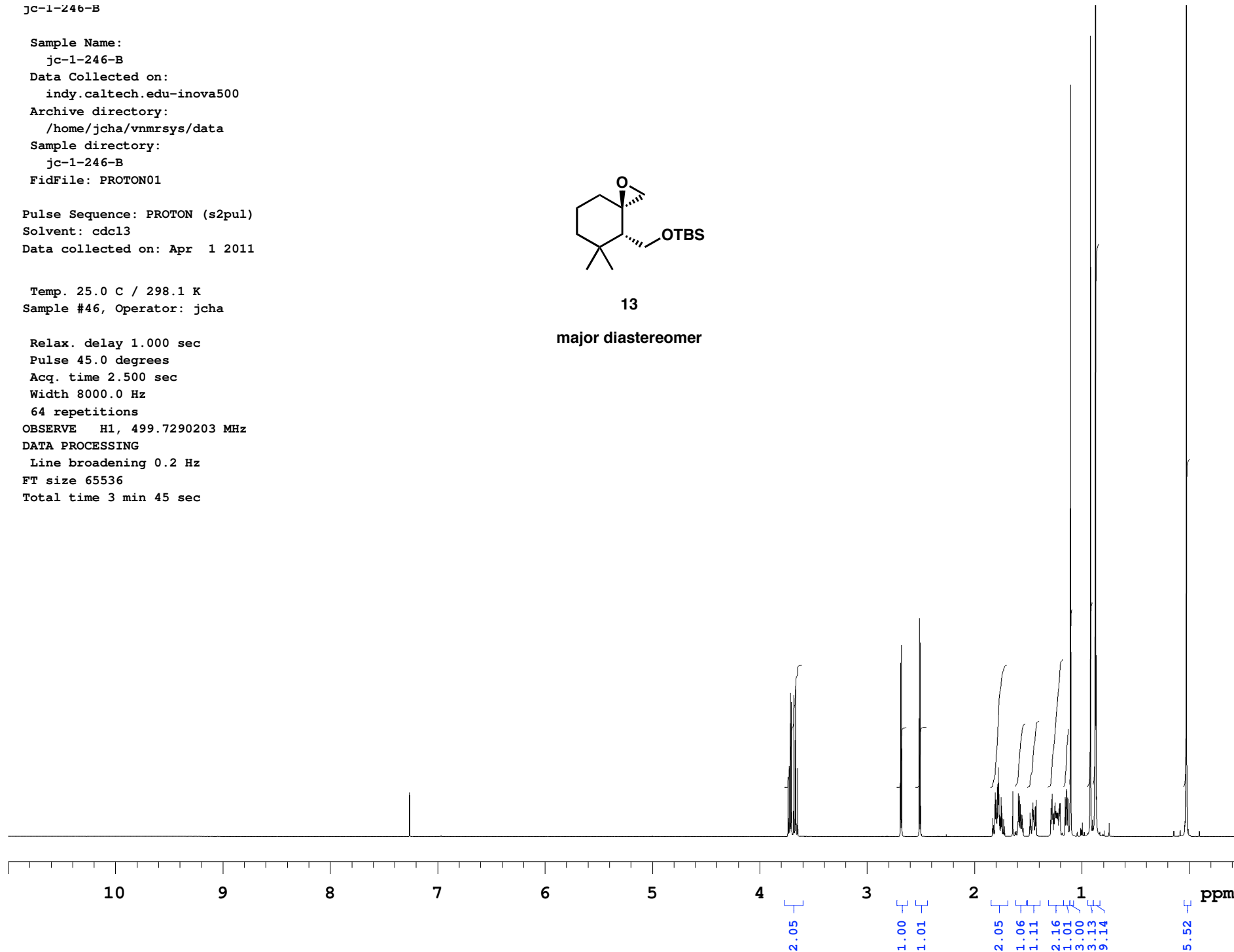
FT size 65536

Total time 3 min 45 sec



13

major diastereomer



jc-1-246-B

Sample Name:

jc-1-246-B

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-246-B

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 1 2011

Temp. 25.0 C / 298.1 K

Sample #46, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

512 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

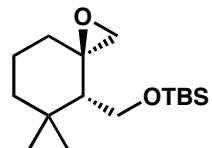
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

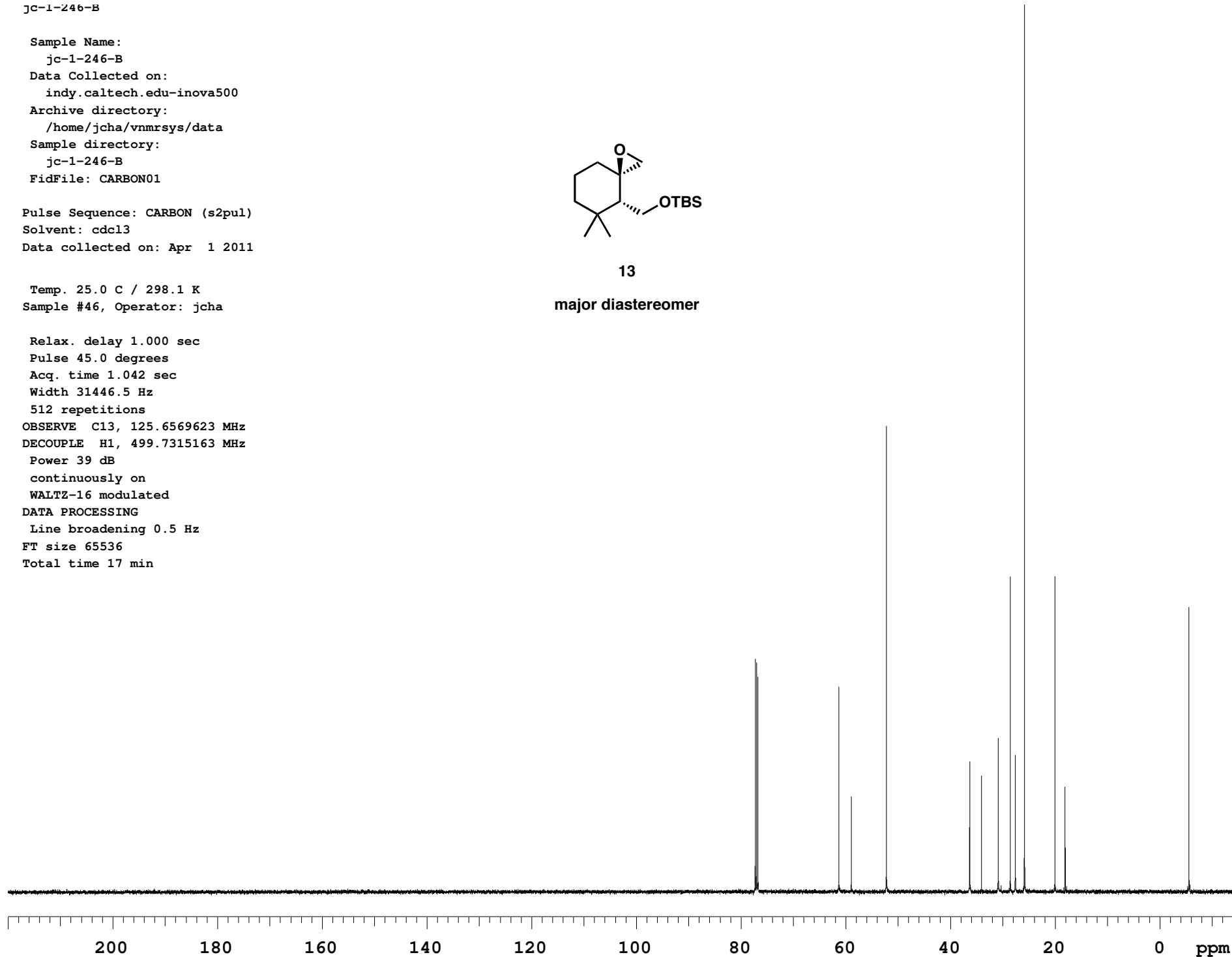
FT size 65536

Total time 17 min



13

major diastereomer



jc-1-246-A

Sample Name:

jc-1-246-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-246-A

FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Apr 4 2011

Temp. 25.0 C / 298.1 K

Sample #46, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

64 repetitions

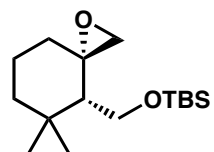
OBSERVE H1, 499.7290200 MHz

DATA PROCESSING

Line broadening 0.2 Hz

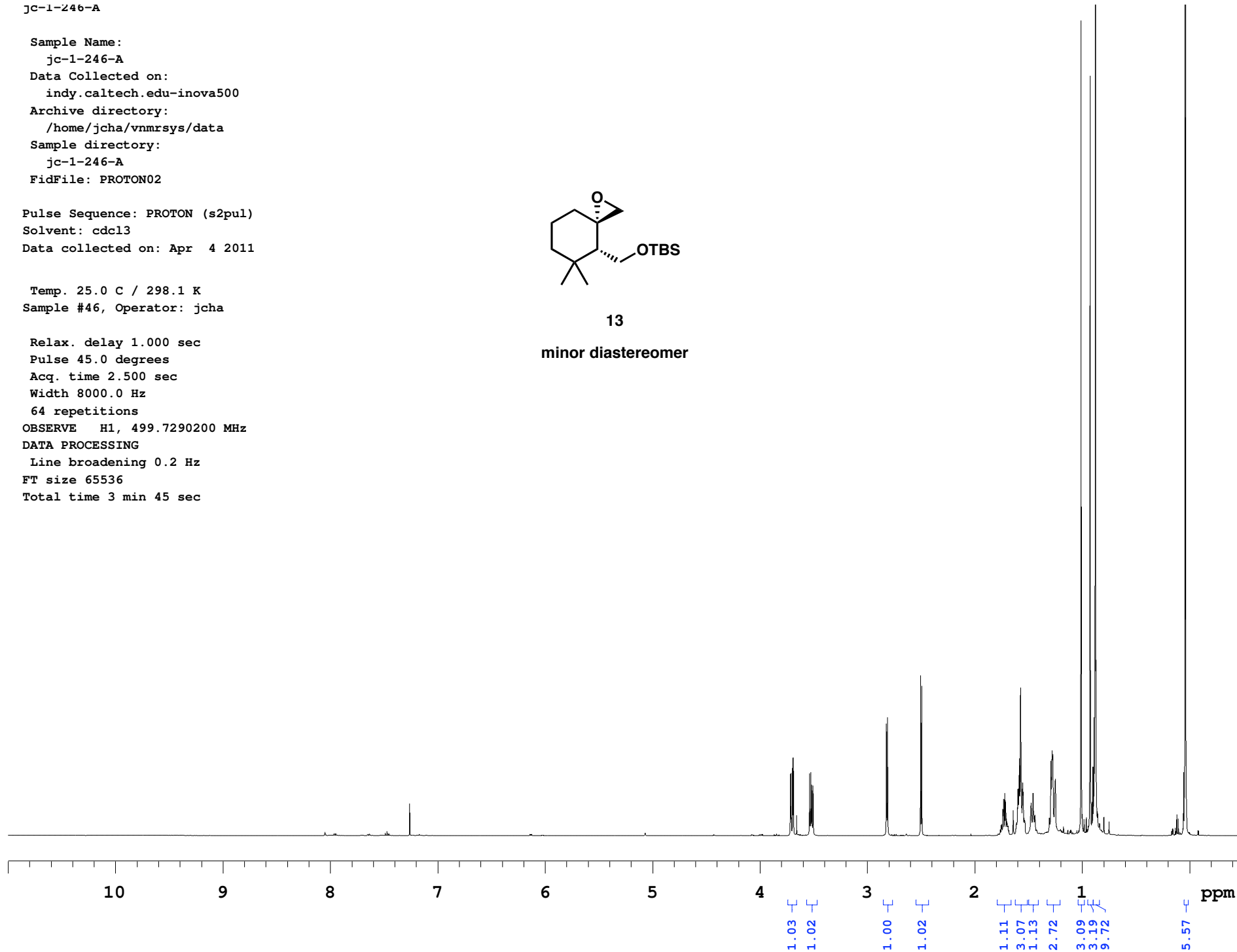
FT size 65536

Total time 3 min 45 sec



13

minor diastereomer



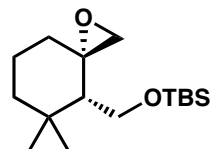
jc-1-246-A

Sample Name:  
jc-1-246-A  
Data Collected on:  
indy.caltech.edu-inova500  
Archive directory:  
/home/jcha/vnmrsys/data  
Sample directory:  
jc-1-246-A  
FidFile: CARBON02

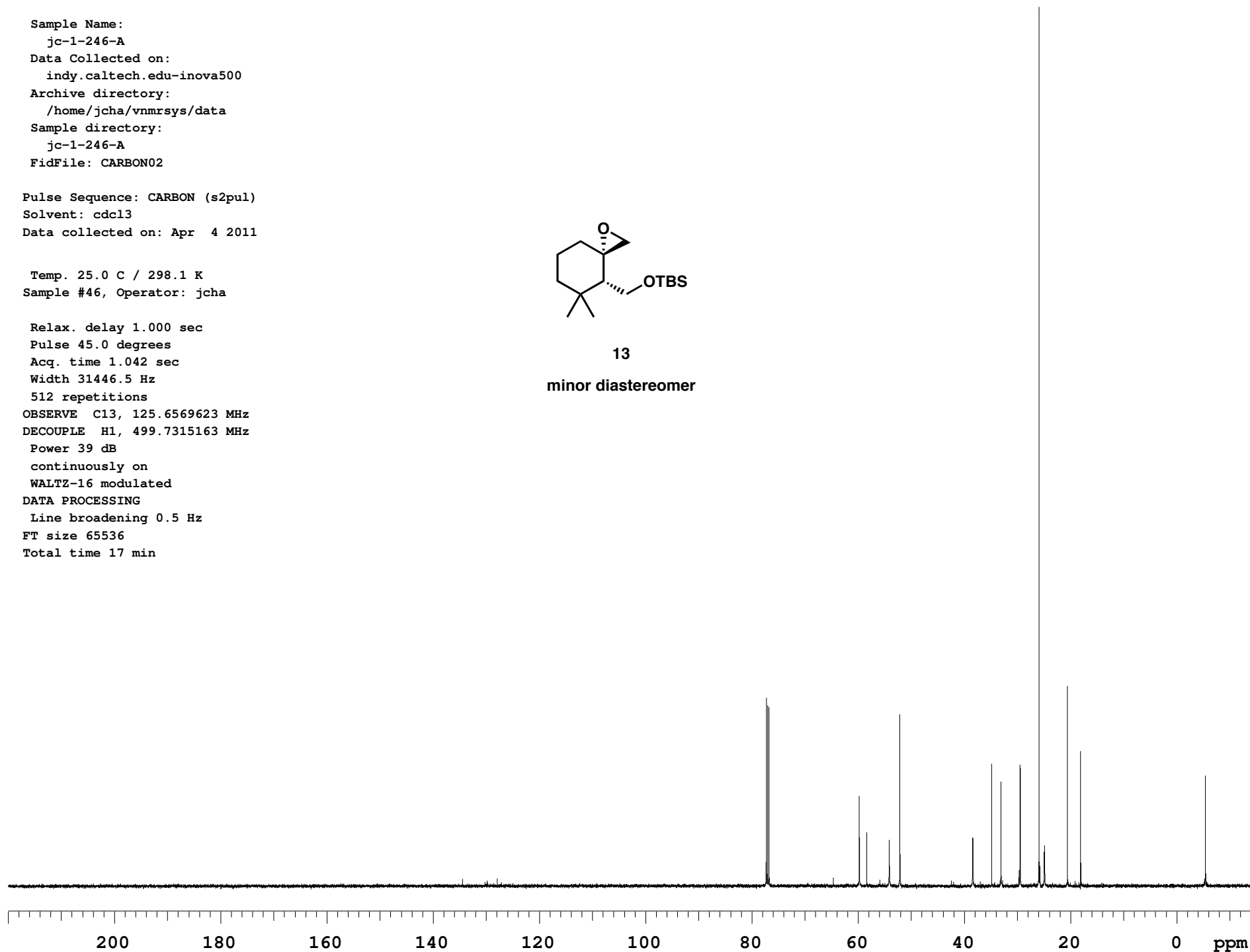
Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Apr 4 2011

Temp. 25.0 C / 298.1 K  
Sample #46, Operator: jcha

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.042 sec  
Width 31446.5 Hz  
512 repetitions  
OBSERVE C13, 125.6569623 MHz  
DECOUPLE H1, 499.7315163 MHz  
Power 39 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 0.5 Hz  
FT size 65536  
Total time 17 min



13  
minor diastereomer



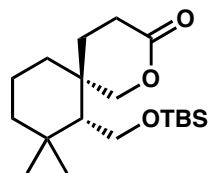
jtsy-v-287

Sample Name:  
jtsy-v-287f15char1  
Data Collected on:  
indy.caltech.edu-inova500  
Archive directory:  
/home/yeomanjo/vnmrsys/data  
Sample directory:  
jtsy-v-287f15char1  
FidFile: PROTON01

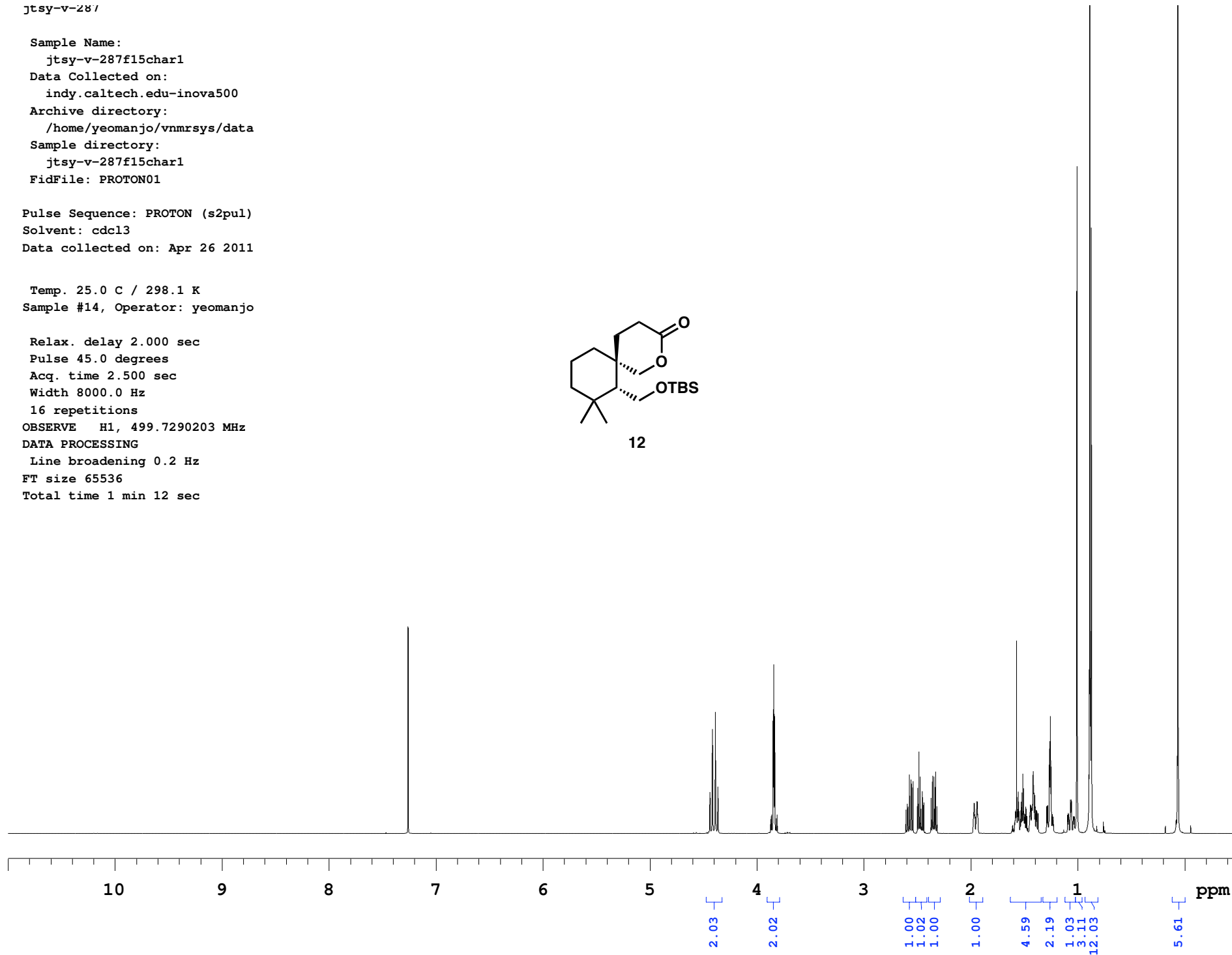
Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Apr 26 2011

Temp. 25.0 C / 298.1 K  
Sample #14, Operator: yeomanjo

Relax. delay 2.000 sec  
Pulse 45.0 degrees  
Acq. time 2.500 sec  
Width 8000.0 Hz  
16 repetitions  
OBSERVE H1, 499.7290203 MHz  
DATA PROCESSING  
Line broadening 0.2 Hz  
FT size 65536  
Total time 1 min 12 sec



12





jtsy-v-28/f15char1

Sample Name:

jtsy-v-287f15char1

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/yeomanjo/vnmrsys/data

Sample directory:

jtsy-v-287f15char1

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 26 2011

Temp. 25.0 C / 298.1 K

Sample #14, Operator: yeomanjo

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1000 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

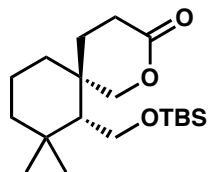
WALTZ-16 modulated

DATA PROCESSING

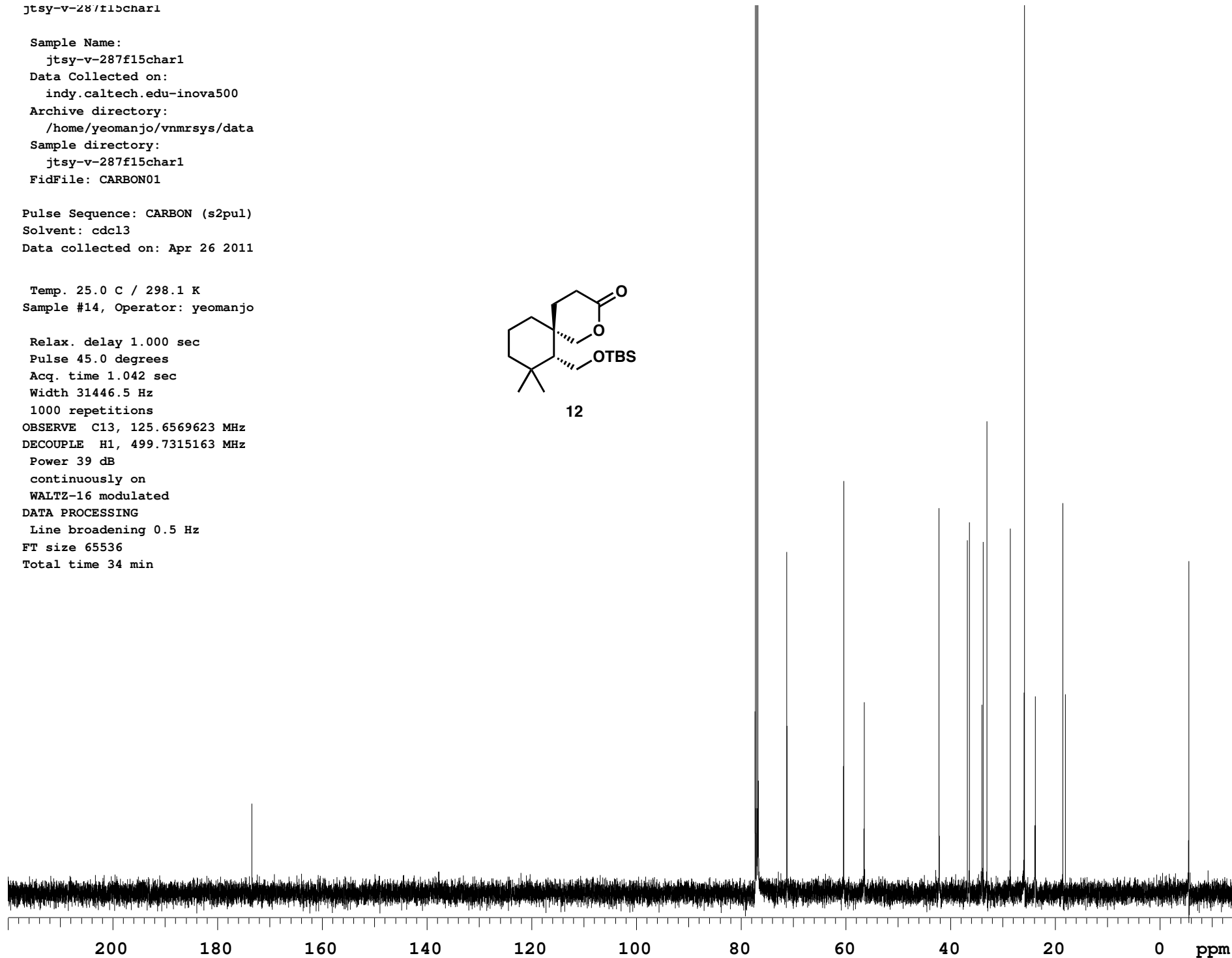
Line broadening 0.5 Hz

FT size 65536

Total time 34 min



12



jtsy-vi-183rf12-15

Sample Name:

jtsy-vi-183rf12-15

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/yeomanjo/vnmrsys/data

Sample directory:

jtsy-vi-183rf12-15

FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jul 21 2011

Sample #14, Operator: yeomanjo

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

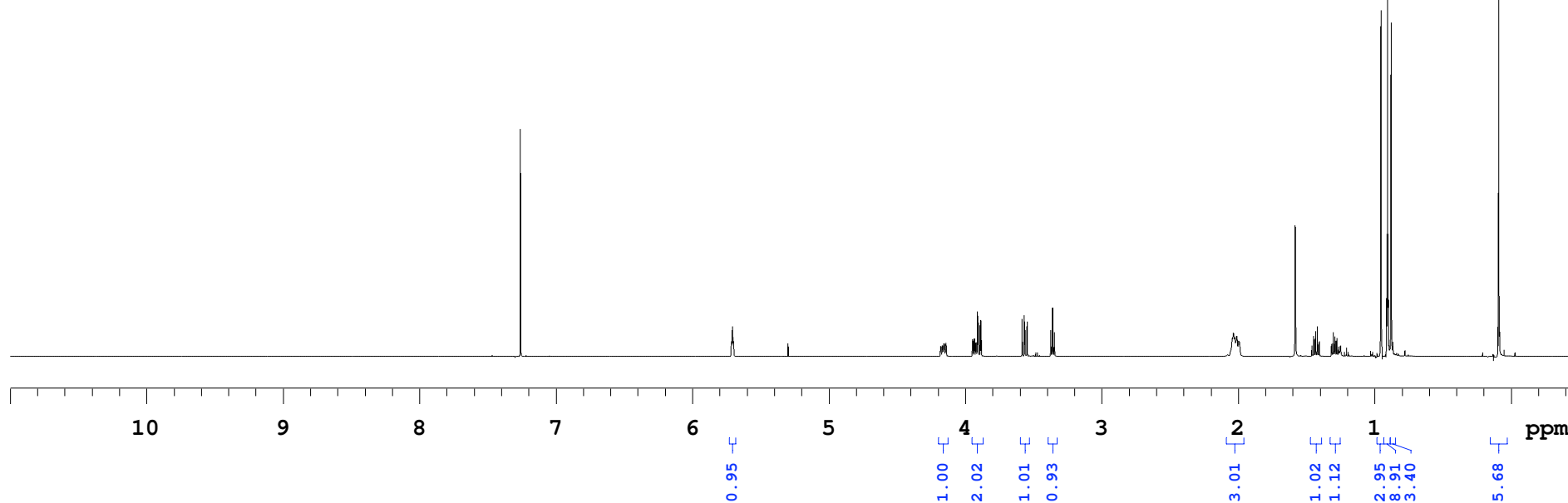
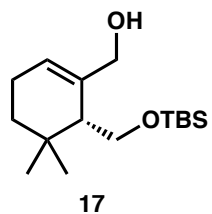
16 repetitions

OBSERVE H1, 499.7225128 MHz

DATA PROCESSING

FT size 65536

Total time 1 min 12 sec



jtsy-vi-183rf12-15

Sample Name:

jtsy-vi-183rf12-15

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/yeomanjo/vnmrsys/data

Sample directory:

jtsy-vi-183rf12-15

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jul 21 2011

Sample #14, Operator: yeomanjo

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1700 repetitions

OBSERVE C13, 125.6553262 MHz

DECOUPLE H1, 499.7250019 MHz

Power 39 dB

continuously on

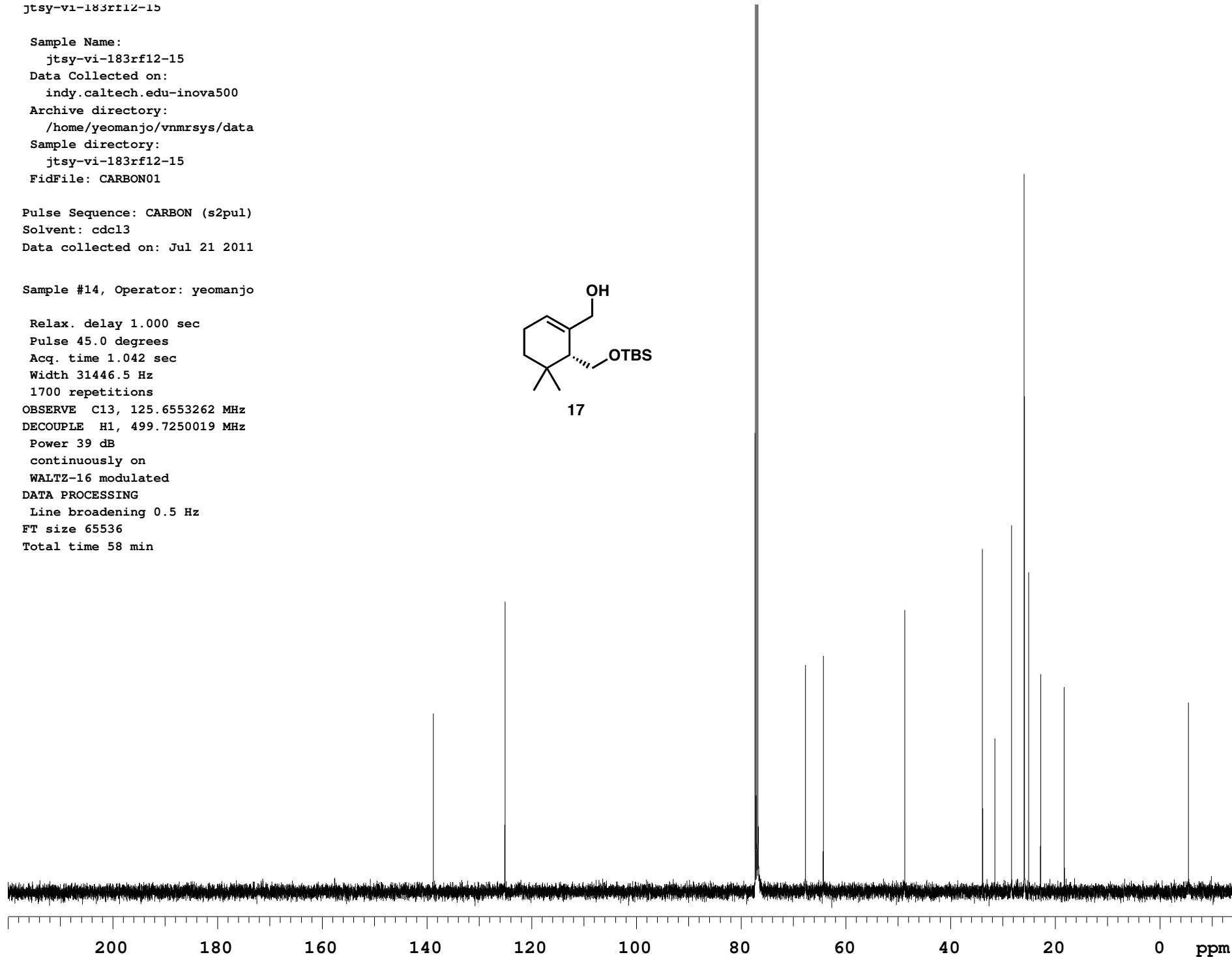
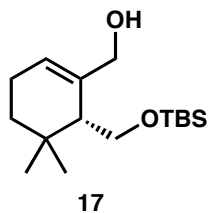
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 58 min



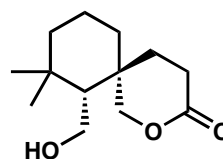
dep-spirolactone-rr

Sample Name:  
dep-spirolactone-rf  
Data Collected on:  
indy.caltech.edu-inova500  
Archive directory:  
/home/yeomanjo/vnmrsys/data  
Sample directory:  
dep-spirolactone-rf  
FidFile: PROTON01

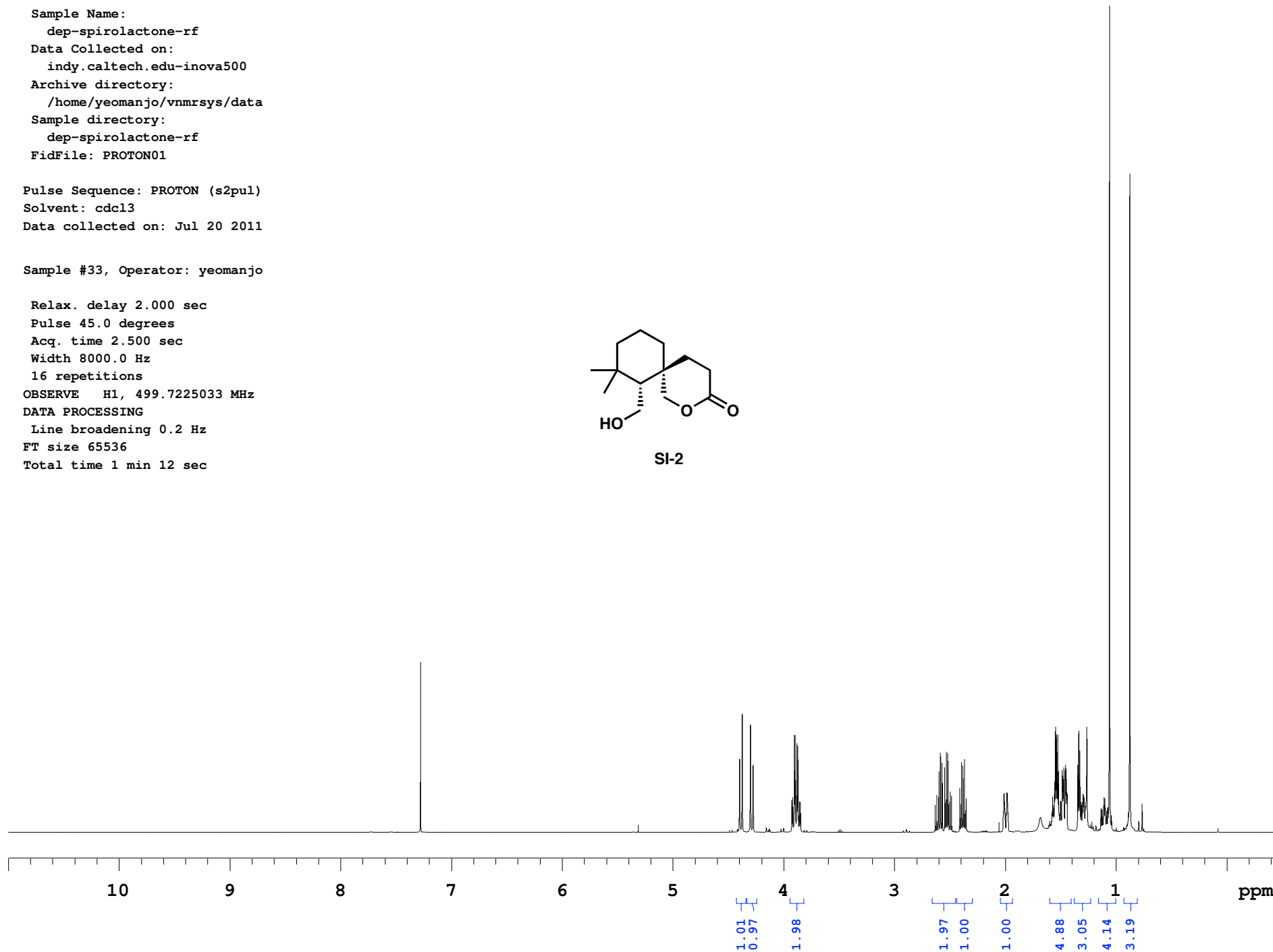
Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Jul 20 2011

Sample #33, Operator: yeomanjo

Relax. delay 2.000 sec  
Pulse 45.0 degrees  
Acq. time 2.500 sec  
Width 8000.0 Hz  
16 repetitions  
OBSERVE H1, 499.7225033 MHz  
DATA PROCESSING  
Line broadening 0.2 Hz  
FT size 65536  
Total time 1 min 12 sec



SI-2



dep-spirolactone-rr

Sample Name:

dep-spirolactone-rf

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/yeomanjo/vnmrsys/data

Sample directory:

dep-spirolactone-rf

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jul 20 2011

Sample #33, Operator: yeomanjo

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1160 repetitions

OBSERVE C13, 125.6553291 MHz

DECOUPLE H1, 499.7250019 MHz

Power 39 dB

continuously on

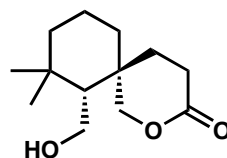
WALTZ-16 modulated

DATA PROCESSING

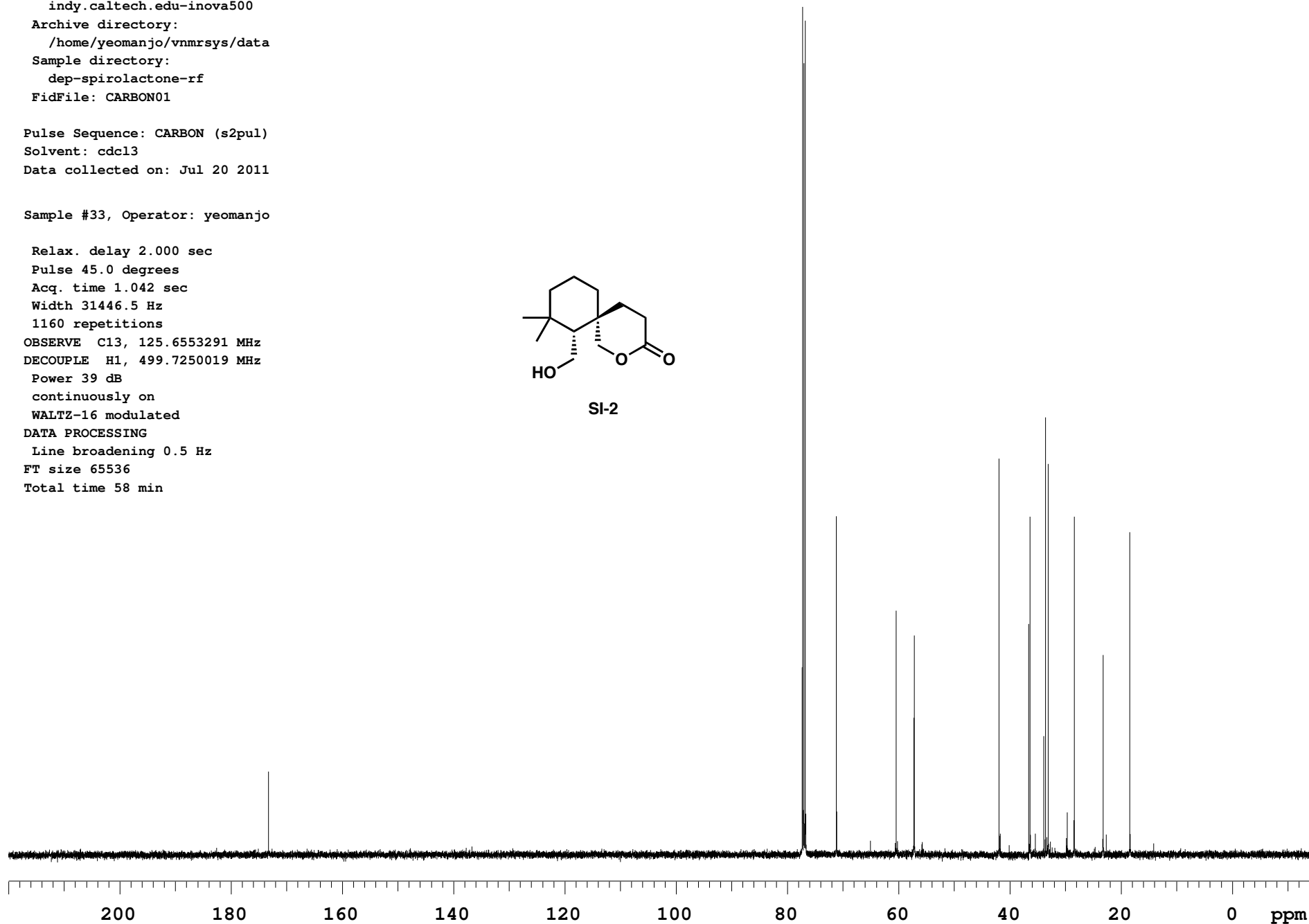
Line broadening 0.5 Hz

FT size 65536

Total time 58 min



SI-2



jc-1-210-A

Sample Name:

jc-1-210-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-210-A

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Apr 8 2011

Temp. 25.0 C / 298.1 K

Sample #41, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

64 repetitions

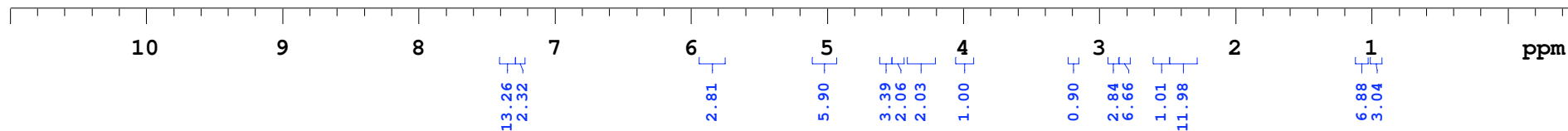
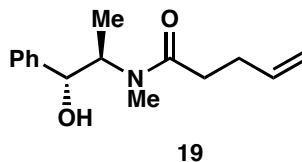
OBSERVE H1, 499.7290121 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 3 min 45 sec



jc-1-210-A

Sample Name:

jc-1-210-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-210-A

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 8 2011

Temp. 25.0 C / 298.1 K

Sample #41, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

512 repetitions

OBSERVE C13, 125.6569729 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

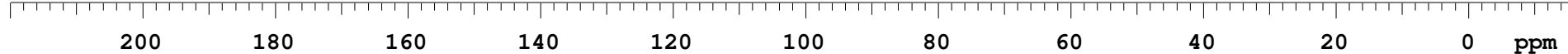
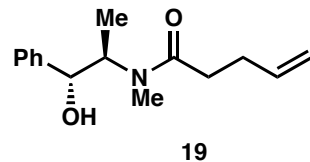
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 17 min



jc-1-231-A

Sample Name:

jc-1-231-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-231-A

FidFile: PROTON03

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Apr 2 2011

Temp. 25.0 C / 298.1 K

Sample #33, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

64 repetitions

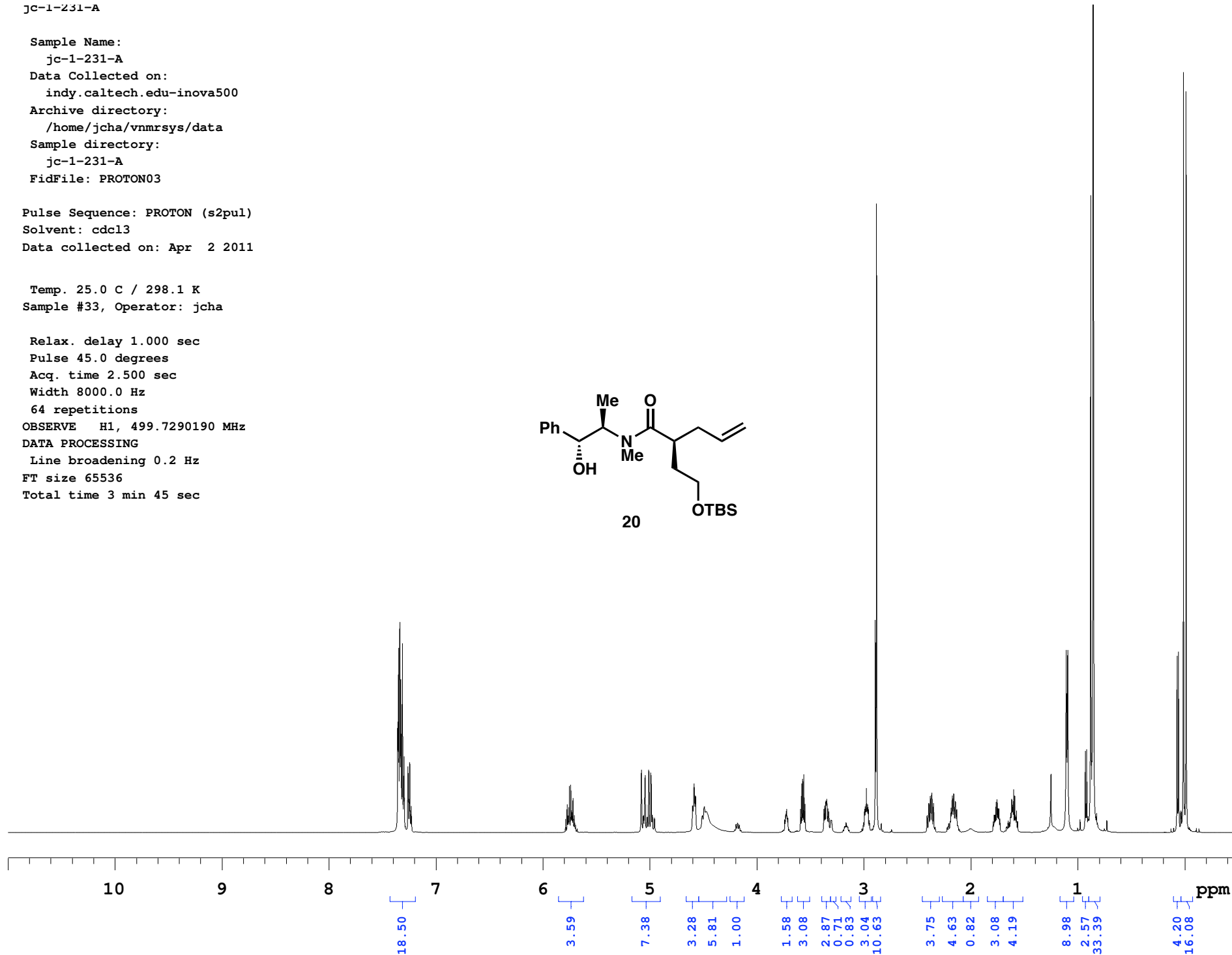
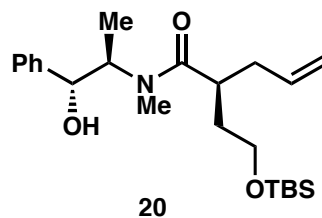
OBSERVE H1, 499.7290190 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 3 min 45 sec





jc-1-231-A

Sample Name:

jc-1-231-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-231-A

FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 2 2011

Temp. 25.0 C / 298.1 K

Sample #33, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

512 repetitions

OBSERVE C13, 125.6569671 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

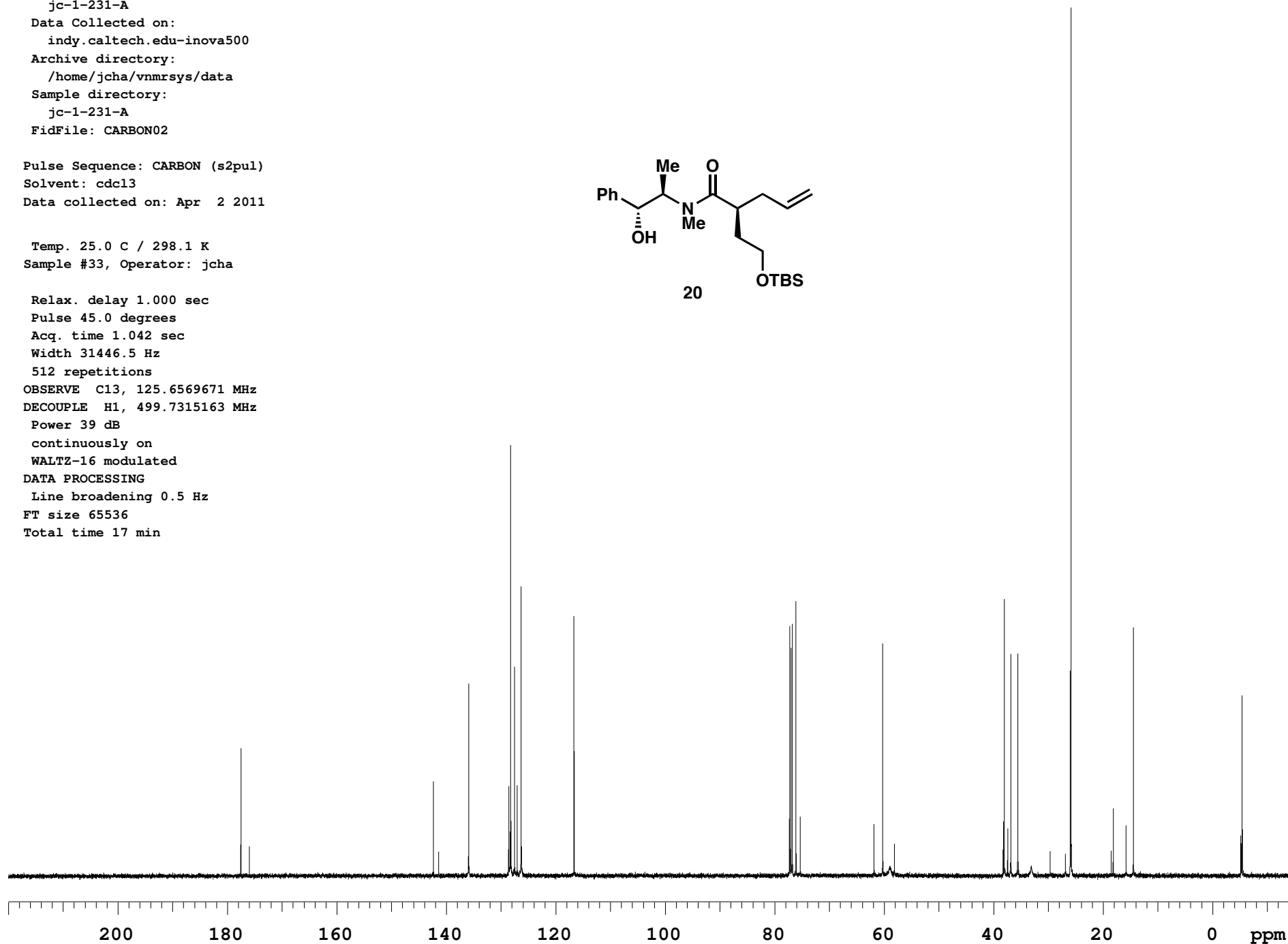
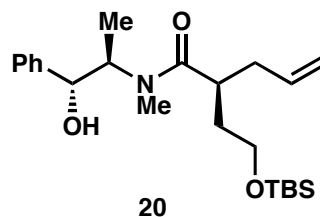
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 17 min



jc-1-241-A

Sample Name:

jc-1-241-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-241-A

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Mar 21 2011

Temp. 25.0 C / 298.1 K

Sample #33, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

32 repetitions

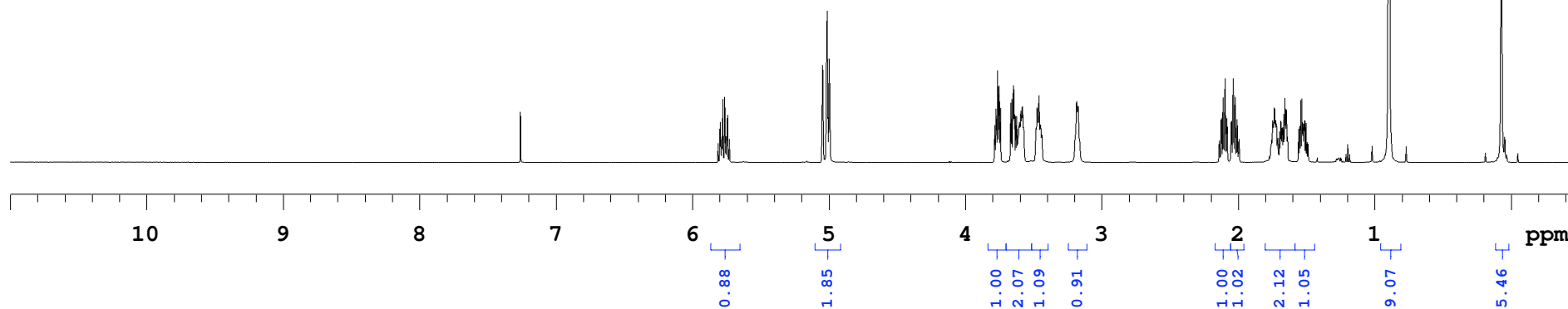
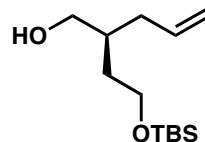
OBSERVE H1, 499.7290203 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min 52 sec



jc-1-241-A-carbon

Sample Name:

jc-1-241-A-carbon

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-241-A-carbon

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Mar 21 2011

Temp. 25.0 C / 298.1 K

Sample #33, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1000 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

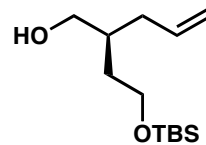
WALTZ-16 modulated

DATA PROCESSING

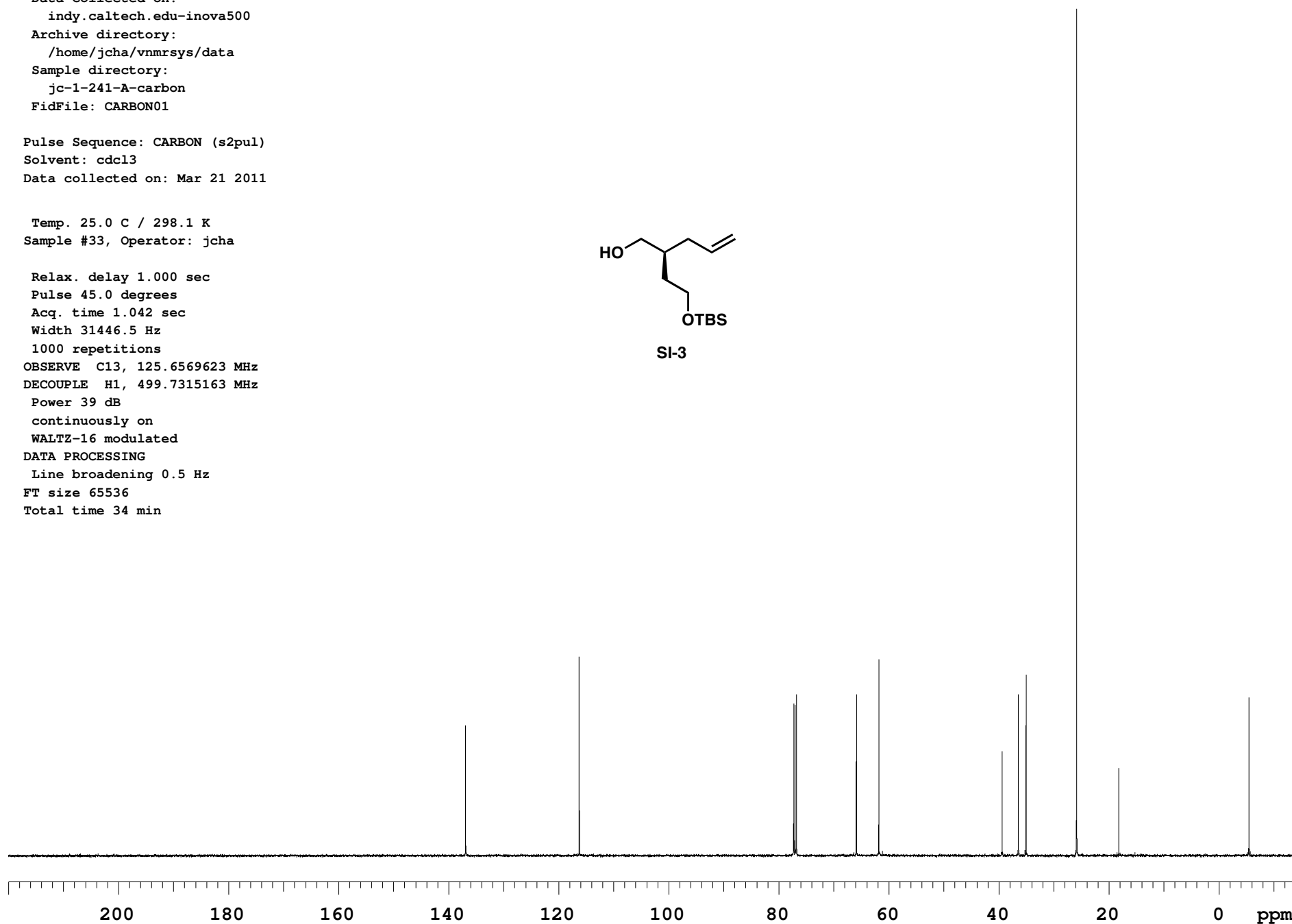
Line broadening 0.5 Hz

FT size 65536

Total time 34 min



SI-3



jc-1-251-A

Sample Name:

jc-1-251-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-251-A

FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Apr 13 2011

Temp. 25.0 C / 298.1 K

Sample #40, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

32 repetitions

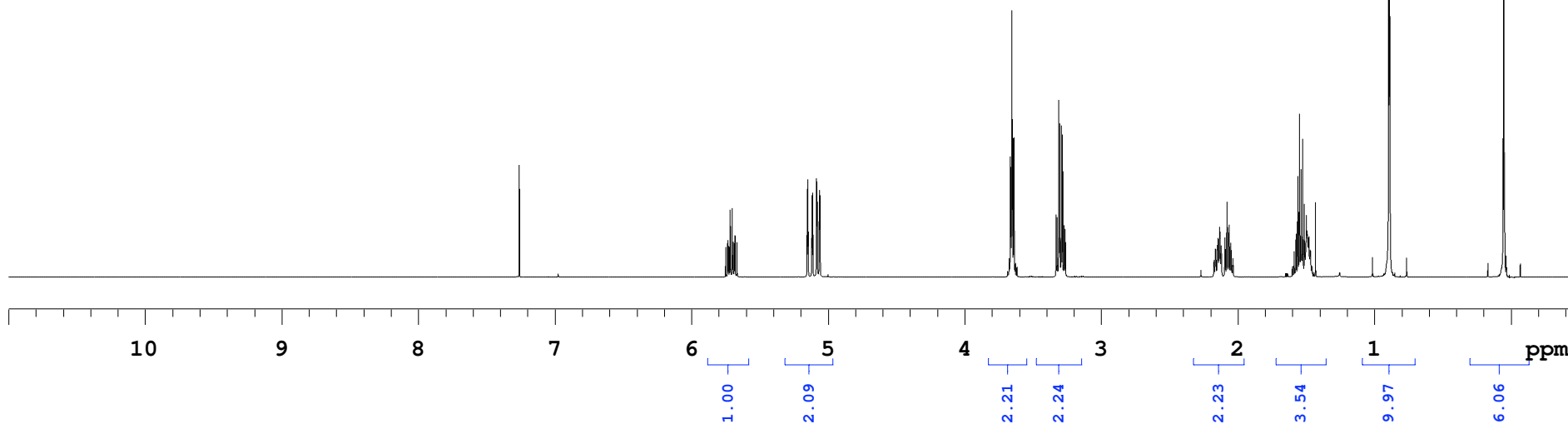
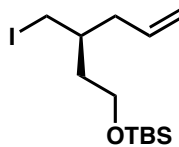
OBSERVE H1, 499.7290203 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 1 min 52 sec



jc-1-251-A

Sample Name:

jc-1-251-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-251-A

FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 13 2011

Temp. 25.0 C / 298.1 K

Sample #40, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

512 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

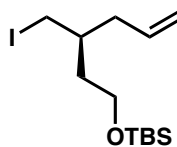
WALTZ-16 modulated

DATA PROCESSING

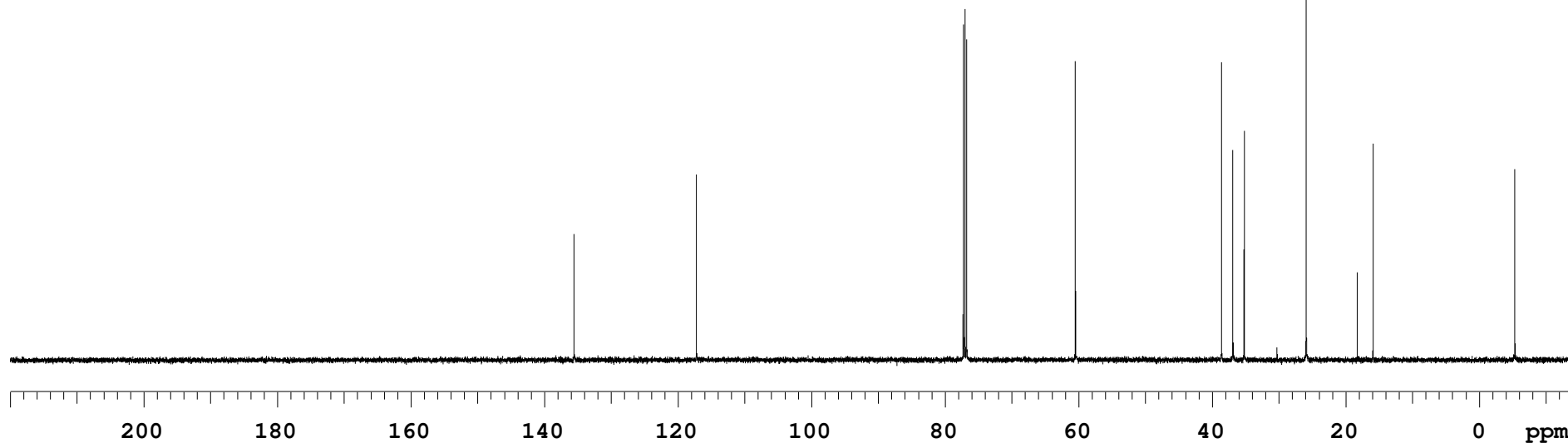
Line broadening 0.5 Hz

FT size 65536

Total time 17 min



21



jc-1-253-A

Sample Name:

jc-1-253-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-253-A

FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jul 20 2011

Sample #1, Operator: jcha

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

16 repetitions

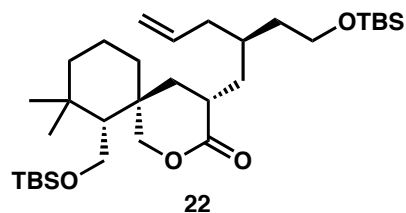
OBSERVE H1, 499.7225125 MHz

DATA PROCESSING

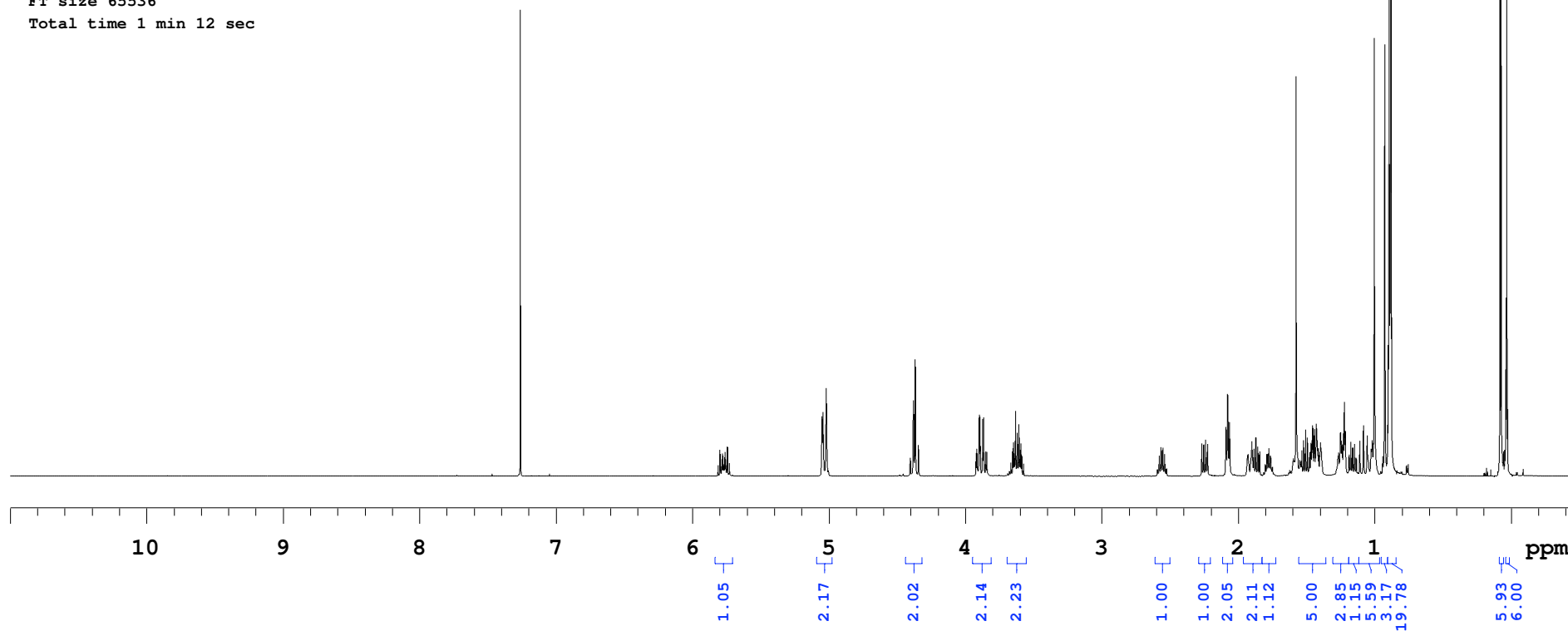
Line broadening 0.2 Hz

FT size 65536

Total time 1 min 12 sec



*less polar  
diastereomer*



jc-1-253-A

Sample Name:

jc-1-253-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-253-A

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jul 20 2011

Sample #1, Operator: jcha

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1000 repetitions

OBSERVE C13, 125.6553262 MHz

DECOUPLE H1, 499.7250019 MHz

Power 39 dB

continuously on

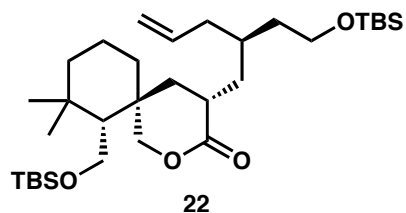
WALTZ-16 modulated

DATA PROCESSING

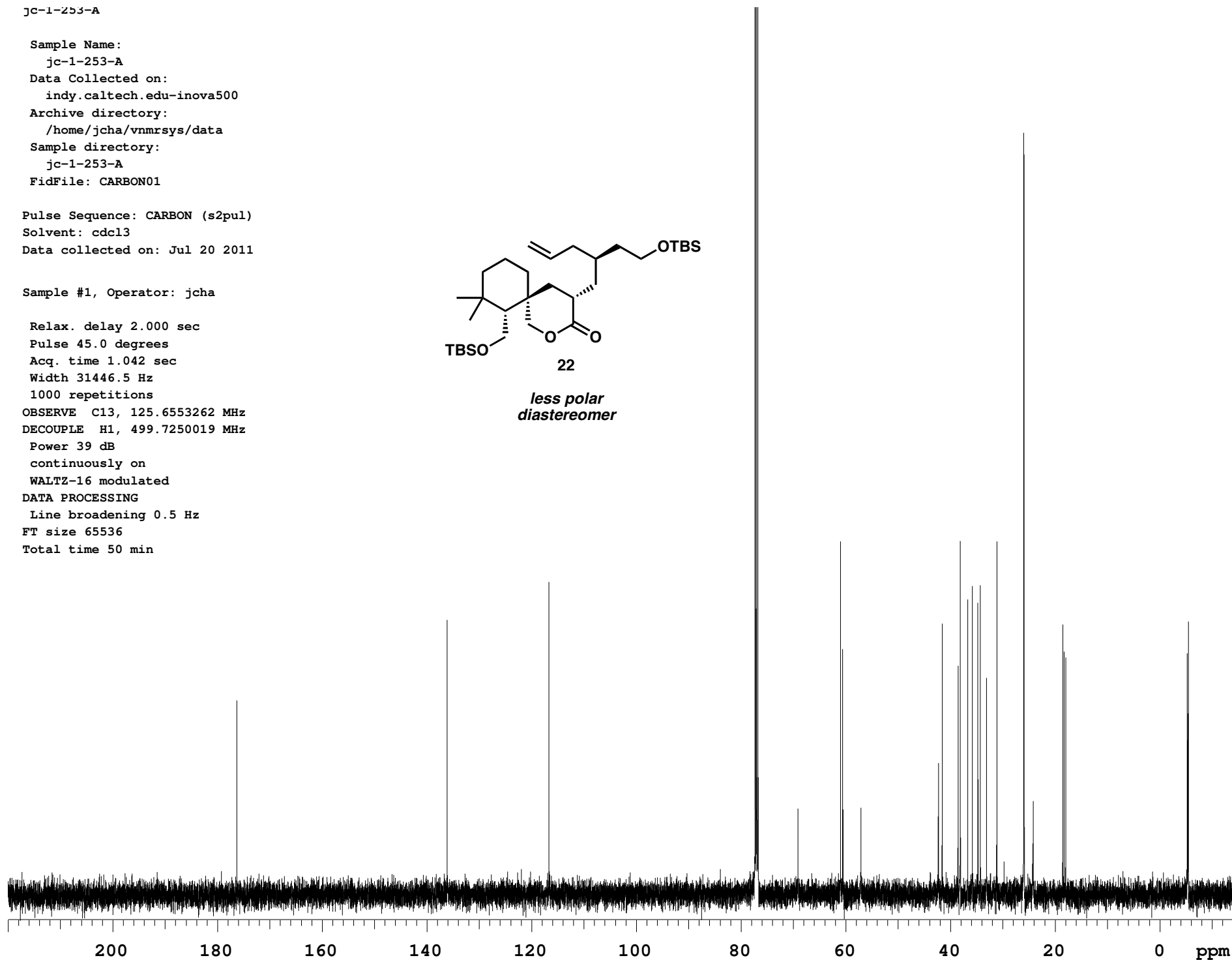
Line broadening 0.5 Hz

FT size 65536

Total time 50 min



*less polar  
diastereomer*



jc-1-253-B

Sample Name:

jc-1-253-B

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-253-B

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Apr 13 2011

Temp. 25.0 C / 298.1 K

Sample #15, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

32 repetitions

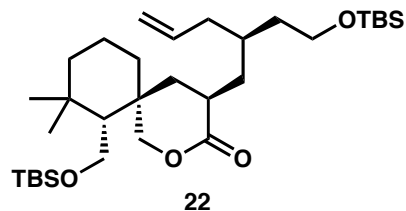
OBSERVE H1, 499.7290203 MHz

DATA PROCESSING

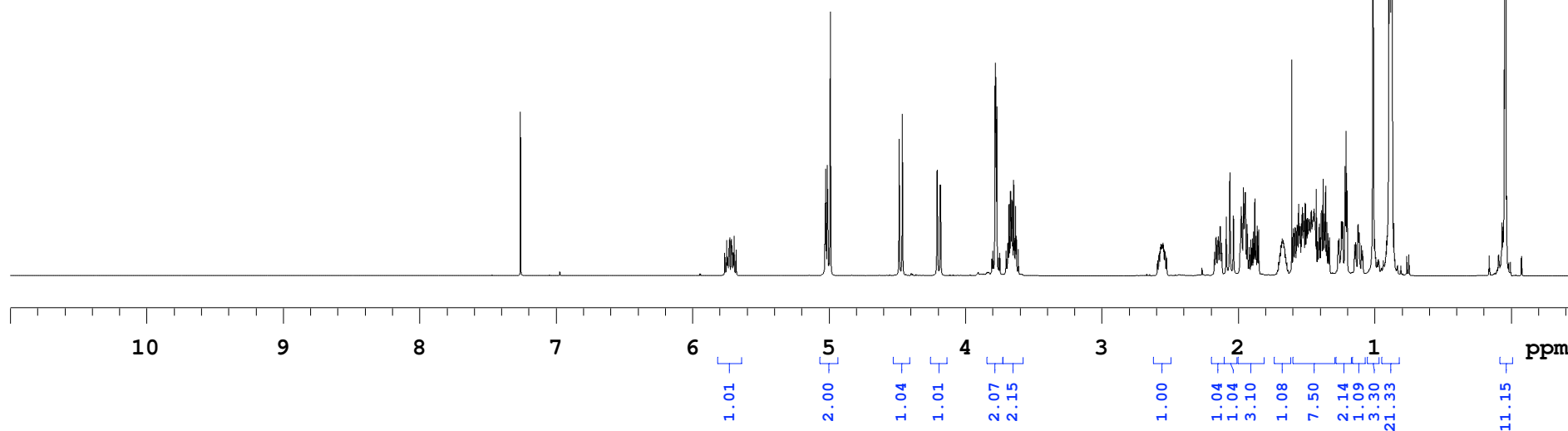
Line broadening 0.2 Hz

FT size 65536

Total time 1 min 52 sec



*more polar  
diastereomer*





jc-1-253-B

Sample Name:

jc-1-253-B

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-253-B

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 13 2011

Temp. 25.0 C / 298.1 K

Sample #15, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

512 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

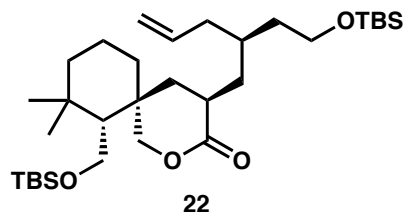
WALTZ-16 modulated

DATA PROCESSING

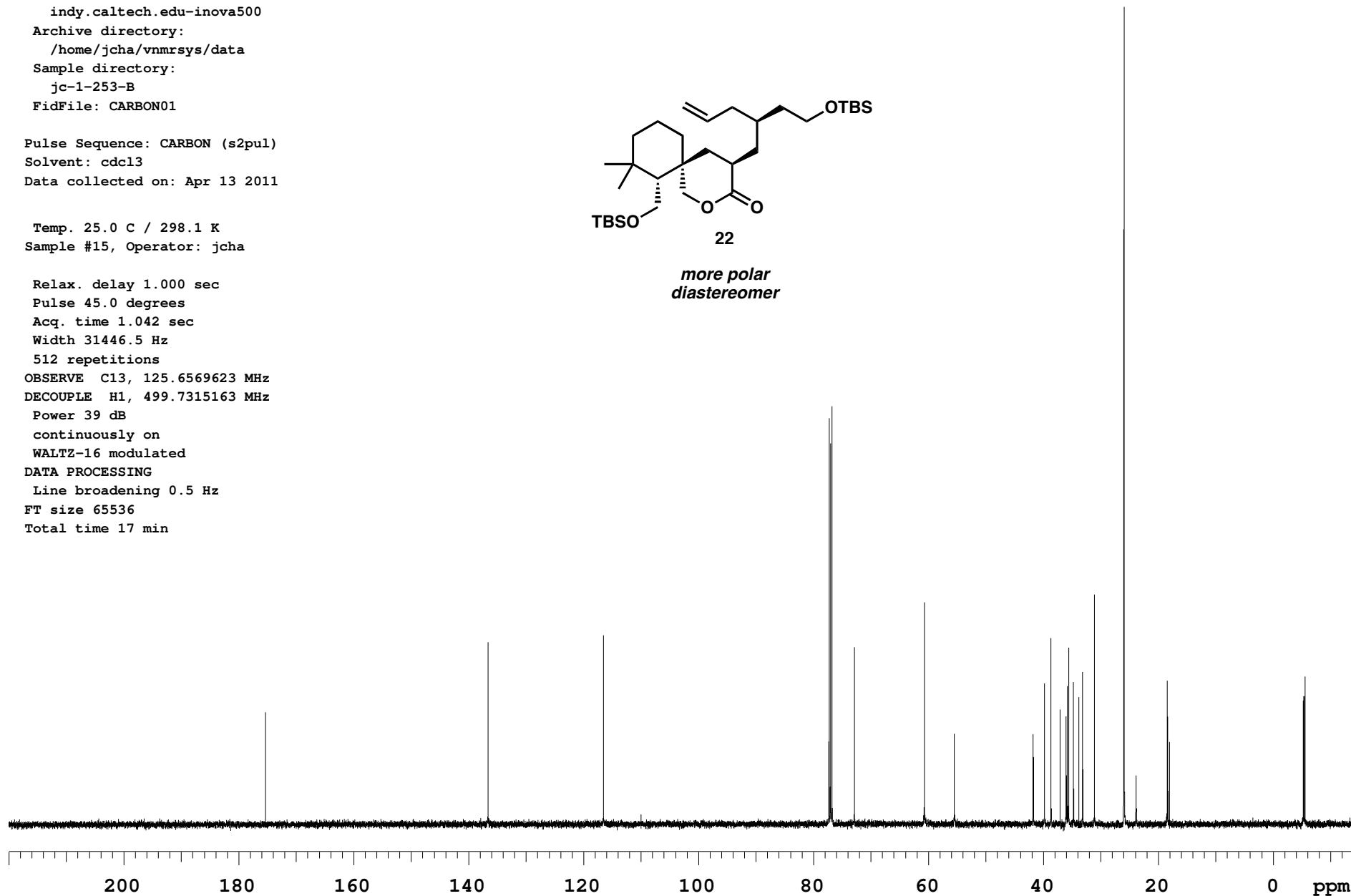
Line broadening 0.5 Hz

FT size 65536

Total time 17 min



*more polar  
diastereomer*



jc-1-254-A

Sample Name:

jc-1-254-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-254-A

FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Apr 17 2011

Temp. 25.0 C / 298.1 K

Sample #15, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

64 repetitions

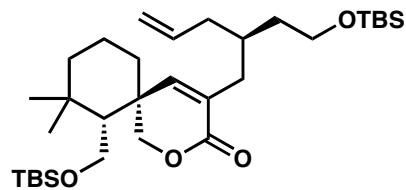
OBSERVE H1, 499.7290200 MHz

DATA PROCESSING

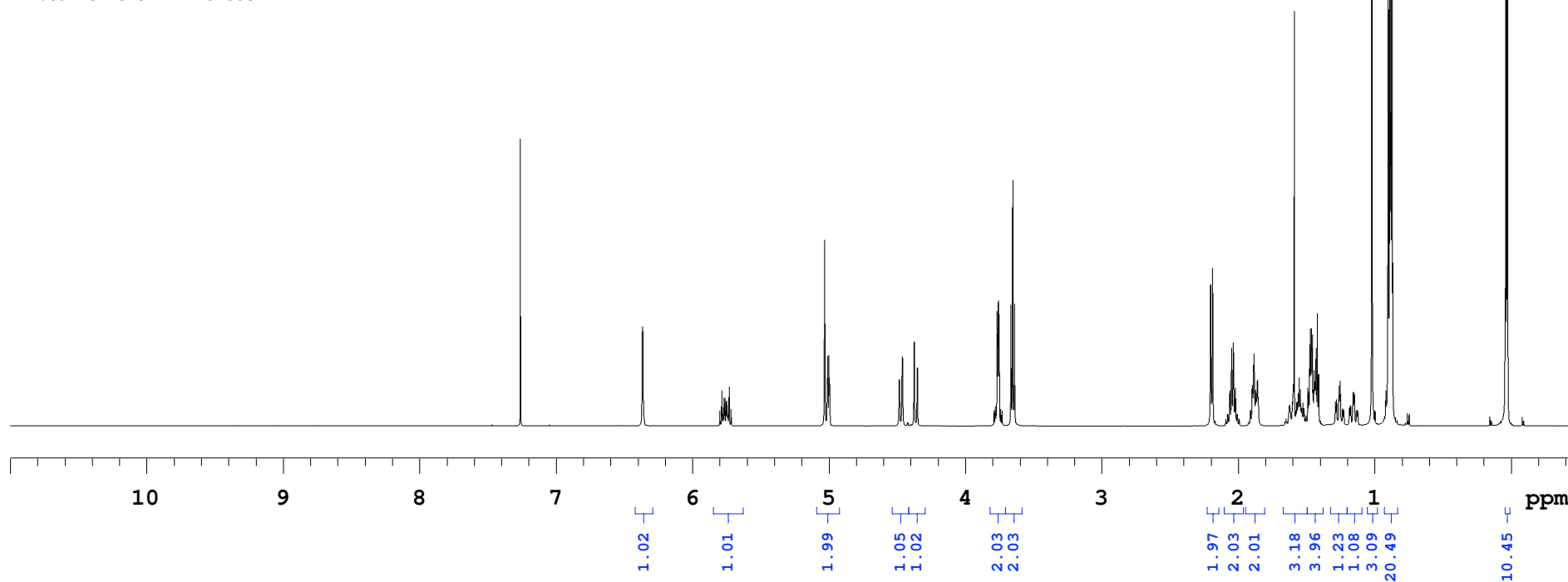
Line broadening 0.2 Hz

FT size 65536

Total time 3 min 45 sec



SI-4



jc-1-254-A

Sample Name:

jc-1-254-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-254-A

FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 17 2011

Temp. 25.0 C / 298.1 K

Sample #15, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1500 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

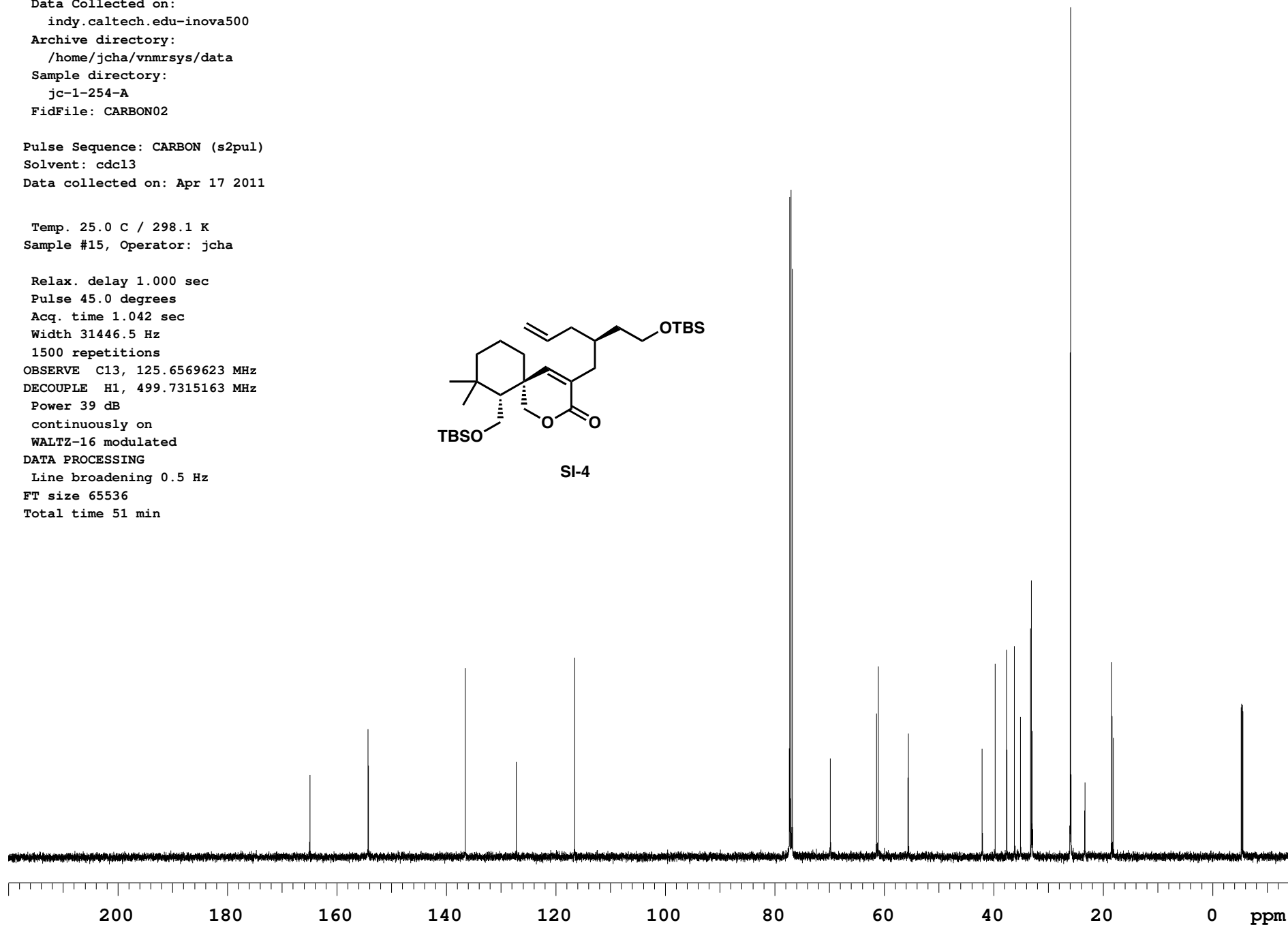
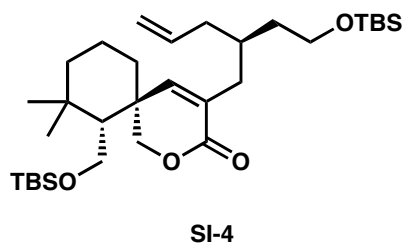
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 51 min



jc-1-256-A

Sample Name:

jc-1-256-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-256-A

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Apr 19 2011

Temp. 25.0 C / 298.1 K

Sample #46, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

64 repetitions

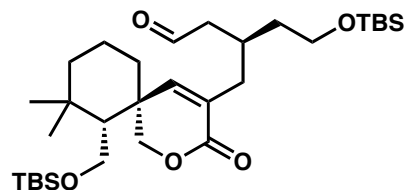
OBSERVE H1, 499.7290208 MHz

DATA PROCESSING

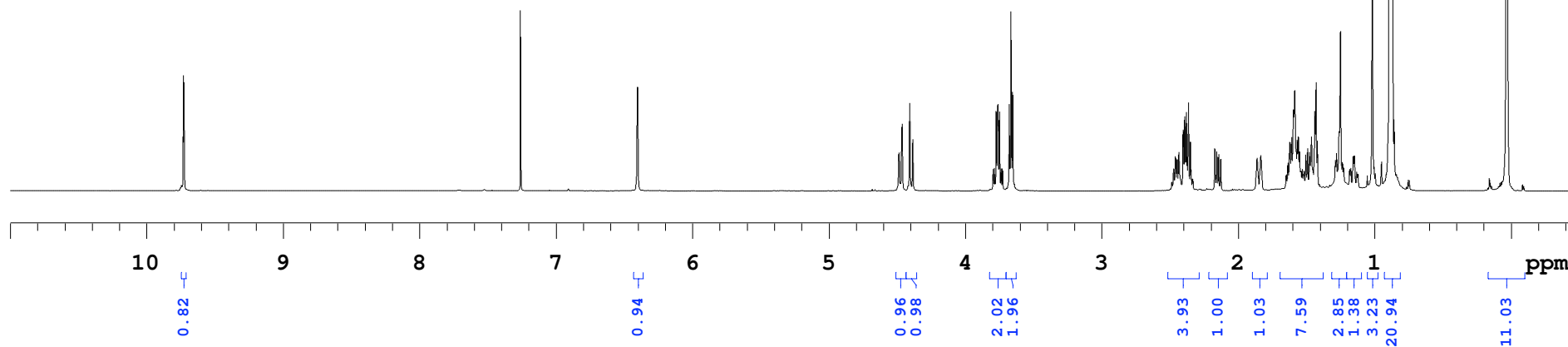
Line broadening 0.2 Hz

FT size 65536

Total time 3 min 45 sec



23



jc-1-256-A

Sample Name:

jc-1-256-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-256-A

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Apr 19 2011

Temp. 25.0 C / 298.1 K

Sample #46, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1500 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

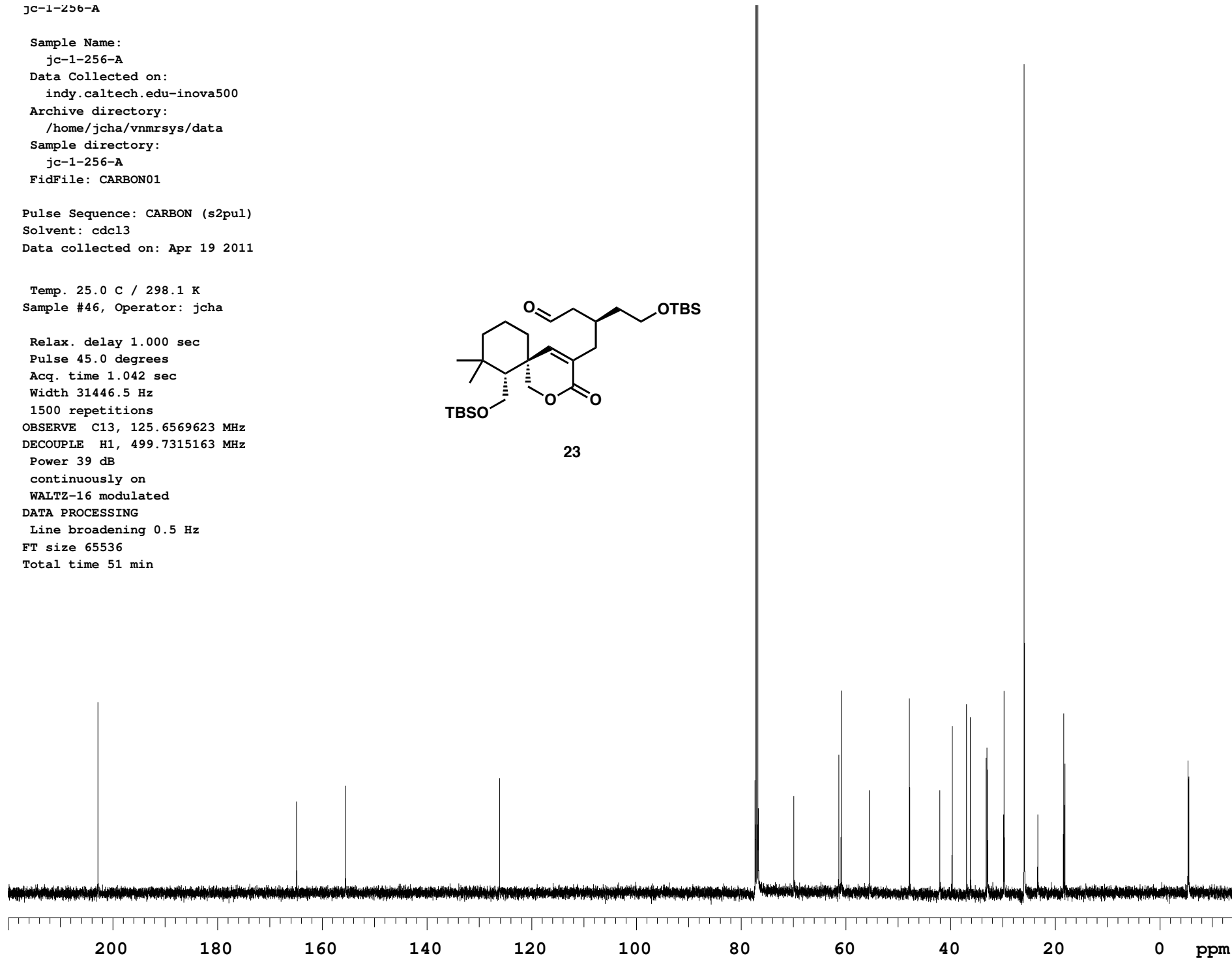
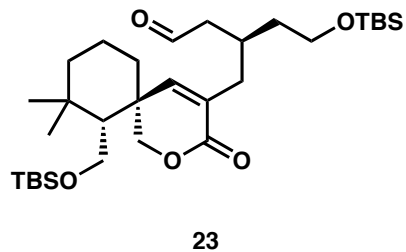
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 51 min



jc-1-257-A

Sample Name:

jc-1-257-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-257-A

FidFile: PROTON04

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jul 10 2011

Sample #2, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

16 repetitions

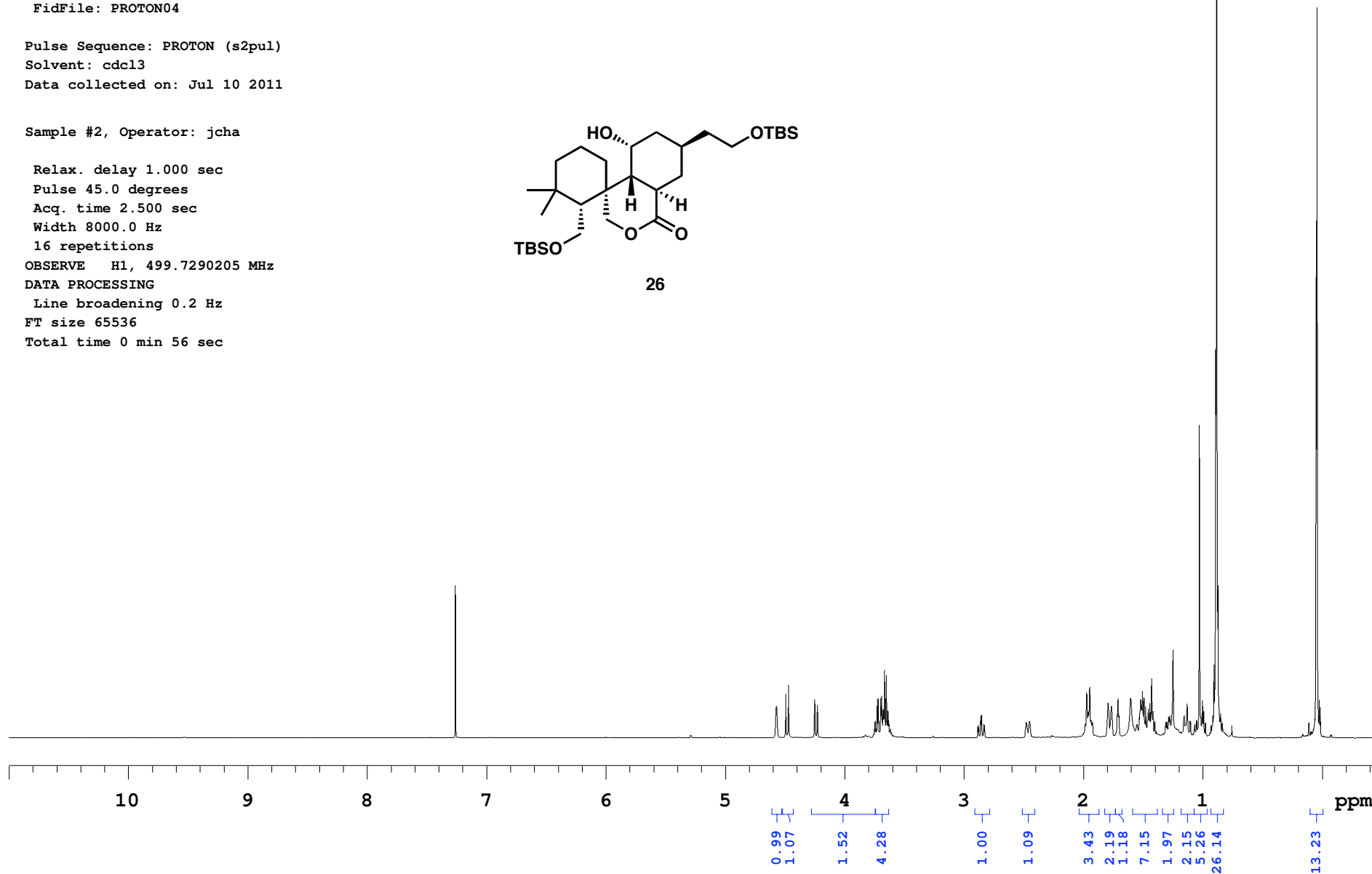
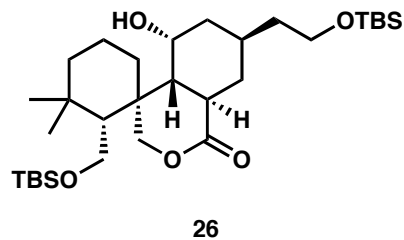
OBSERVE H1, 499.7290205 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 0 min 56 sec



jc-1-257-A

Sample Name:

jc-1-257-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-257-A

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jul 10 2011

Sample #2, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1250 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

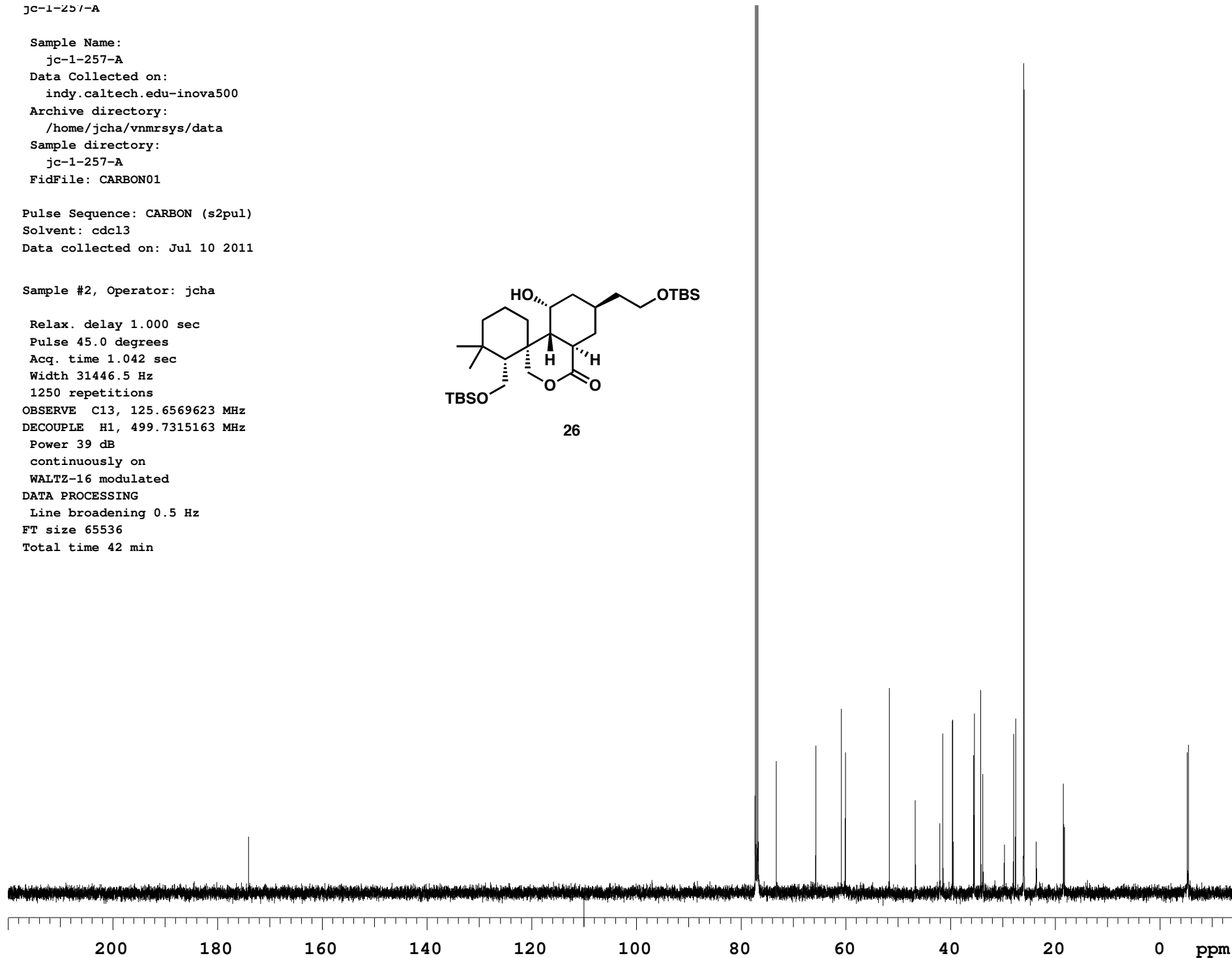
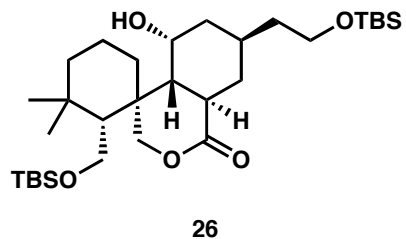
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 42 min



jc-1-258-A

Sample Name:

jc-1-258-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-258-A

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: May 3 2011

Temp. 25.0 C / 298.1 K

Sample #7, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

128 repetitions

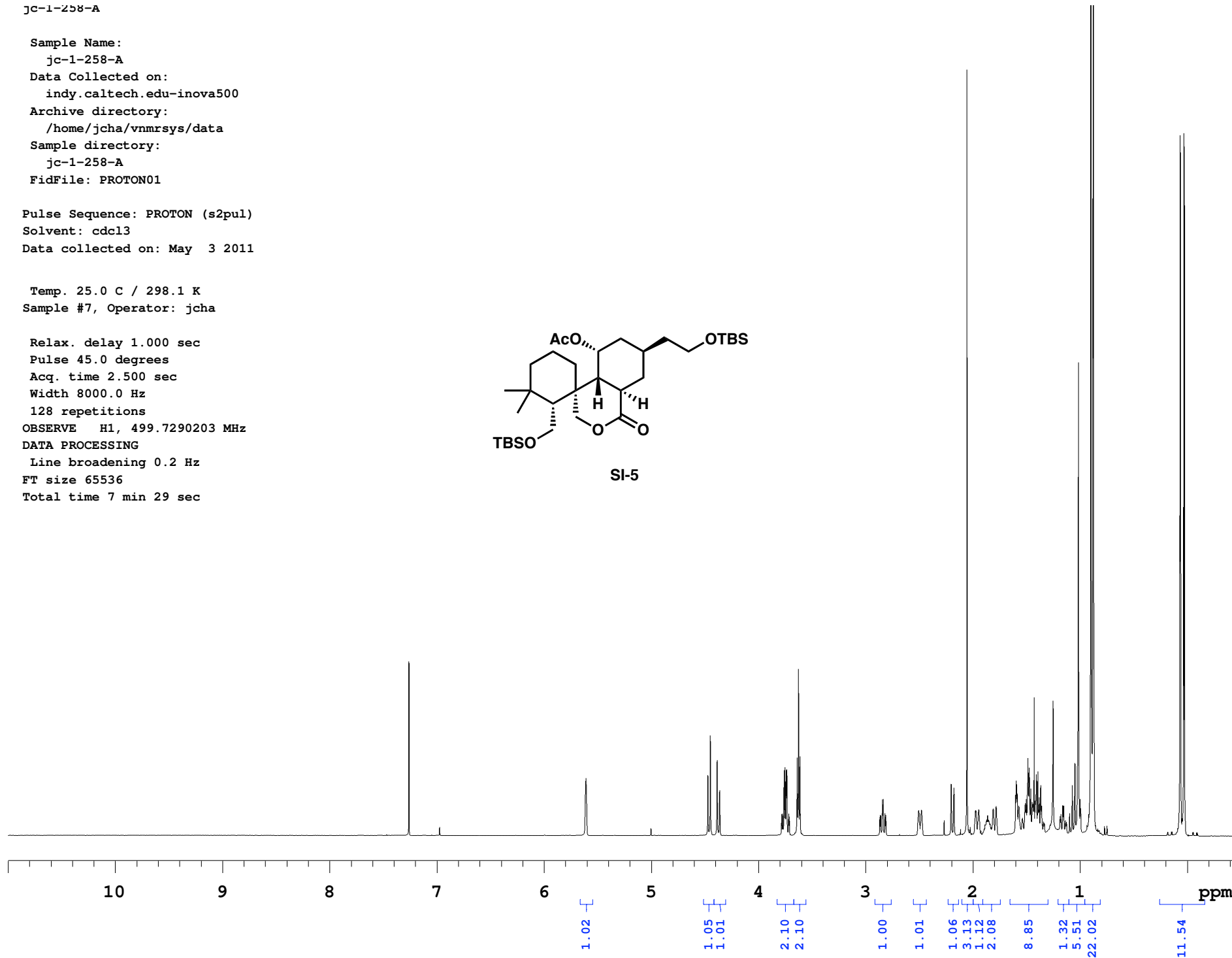
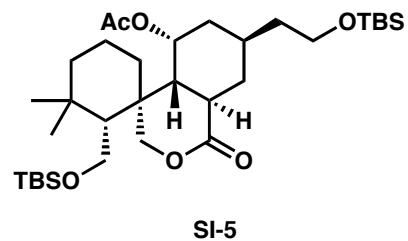
OBSERVE H1, 499.7290203 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 7 min 29 sec





jc-1-258-A

Sample Name:

jc-1-258-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-258-A

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: May 3 2011

Temp. 25.0 C / 298.1 K

Sample #7, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1000 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

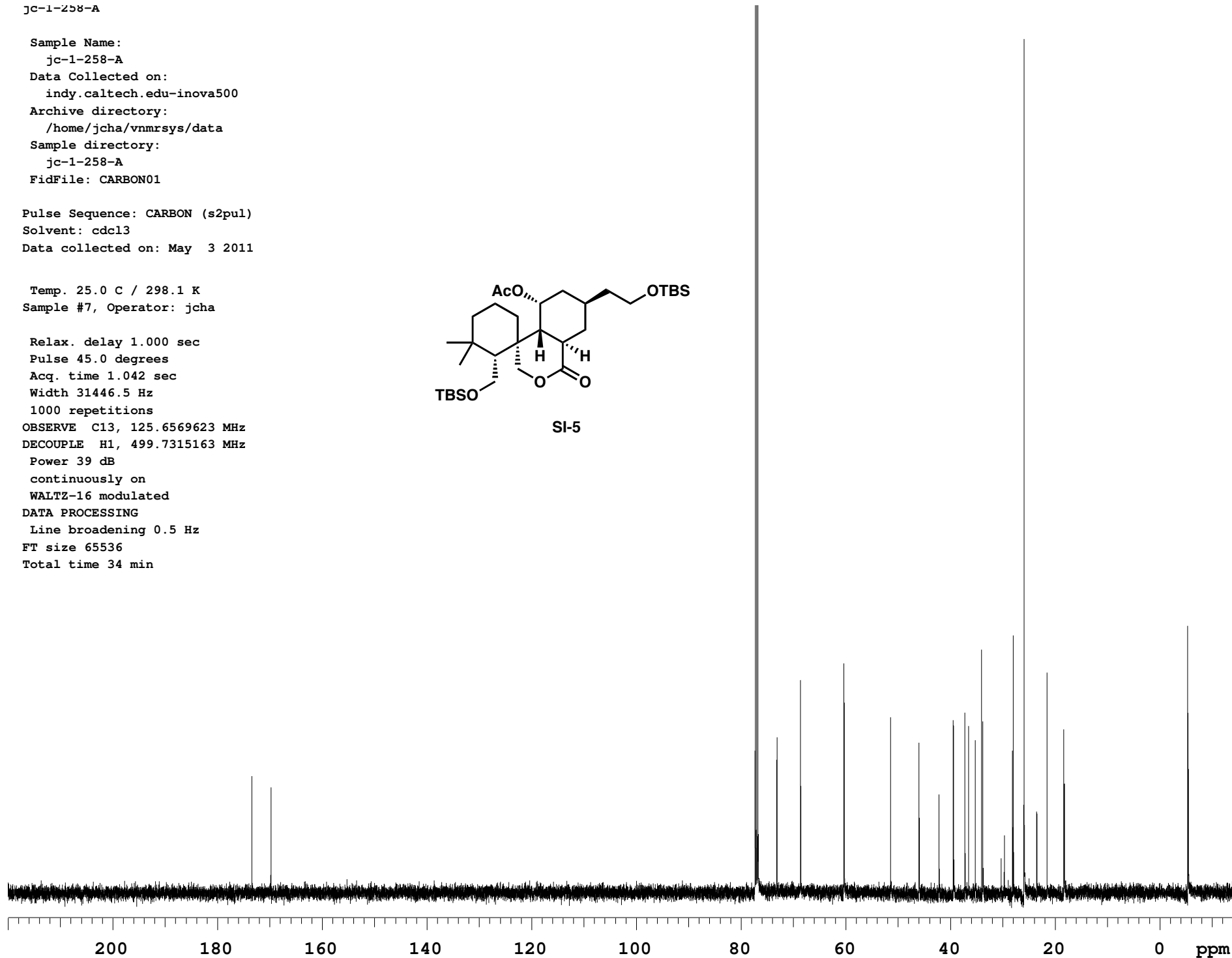
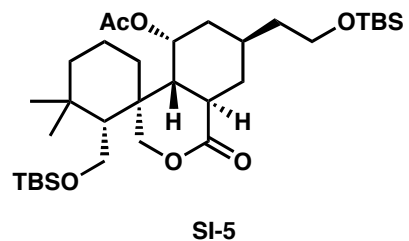
WALTZ-16 modulated

DATA PROCESSING

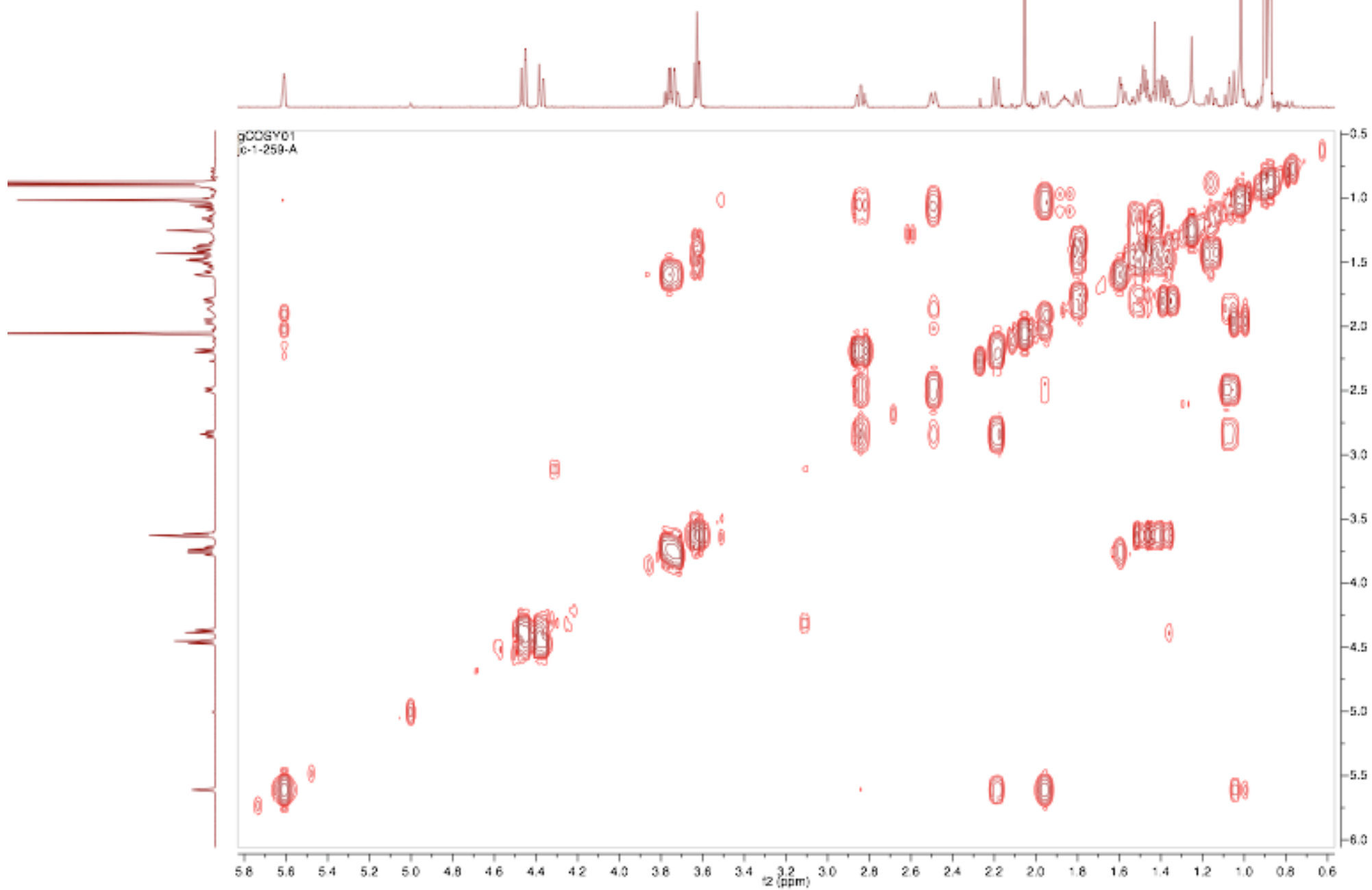
Line broadening 0.5 Hz

FT size 65536

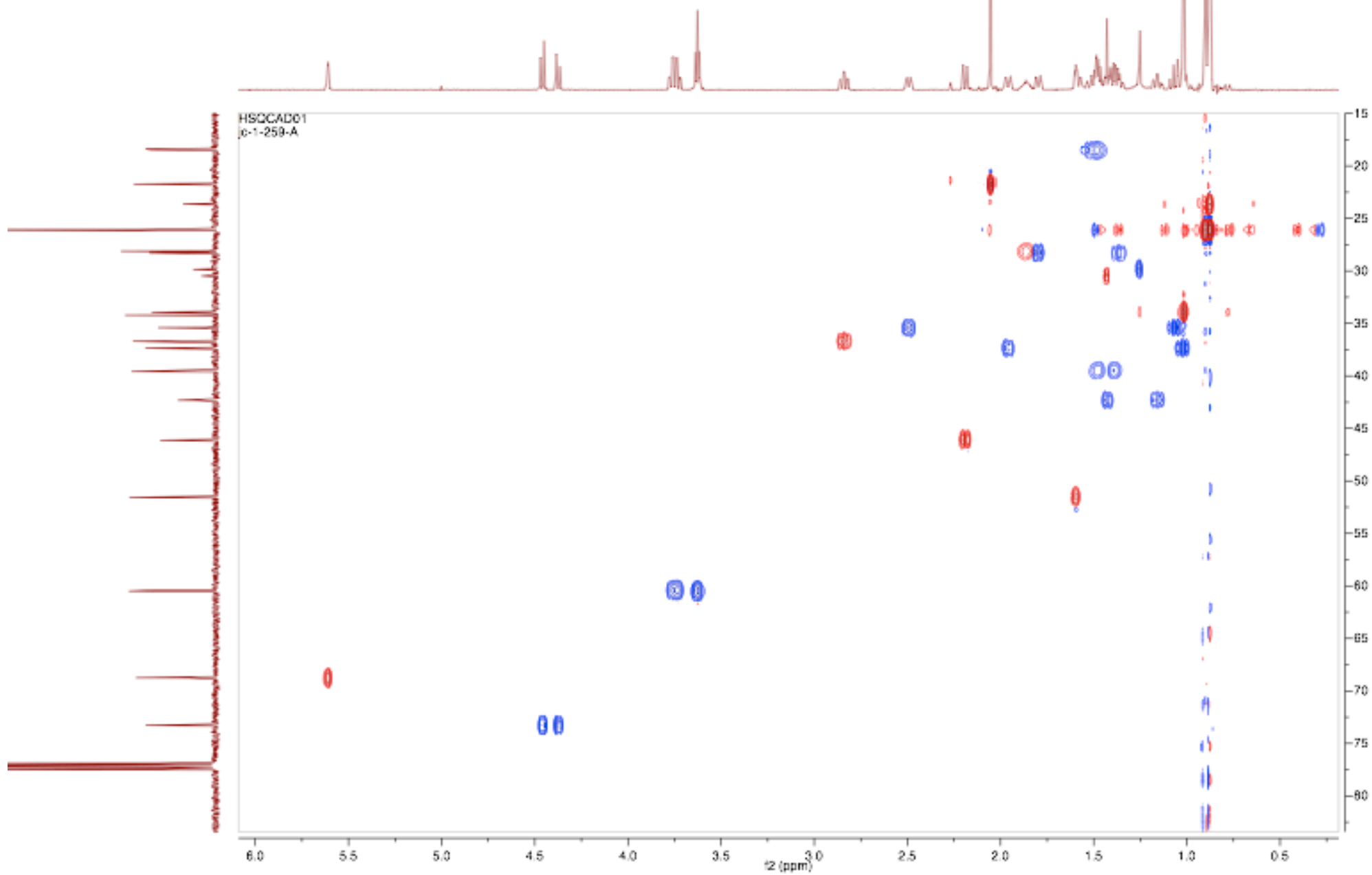
Total time 34 min



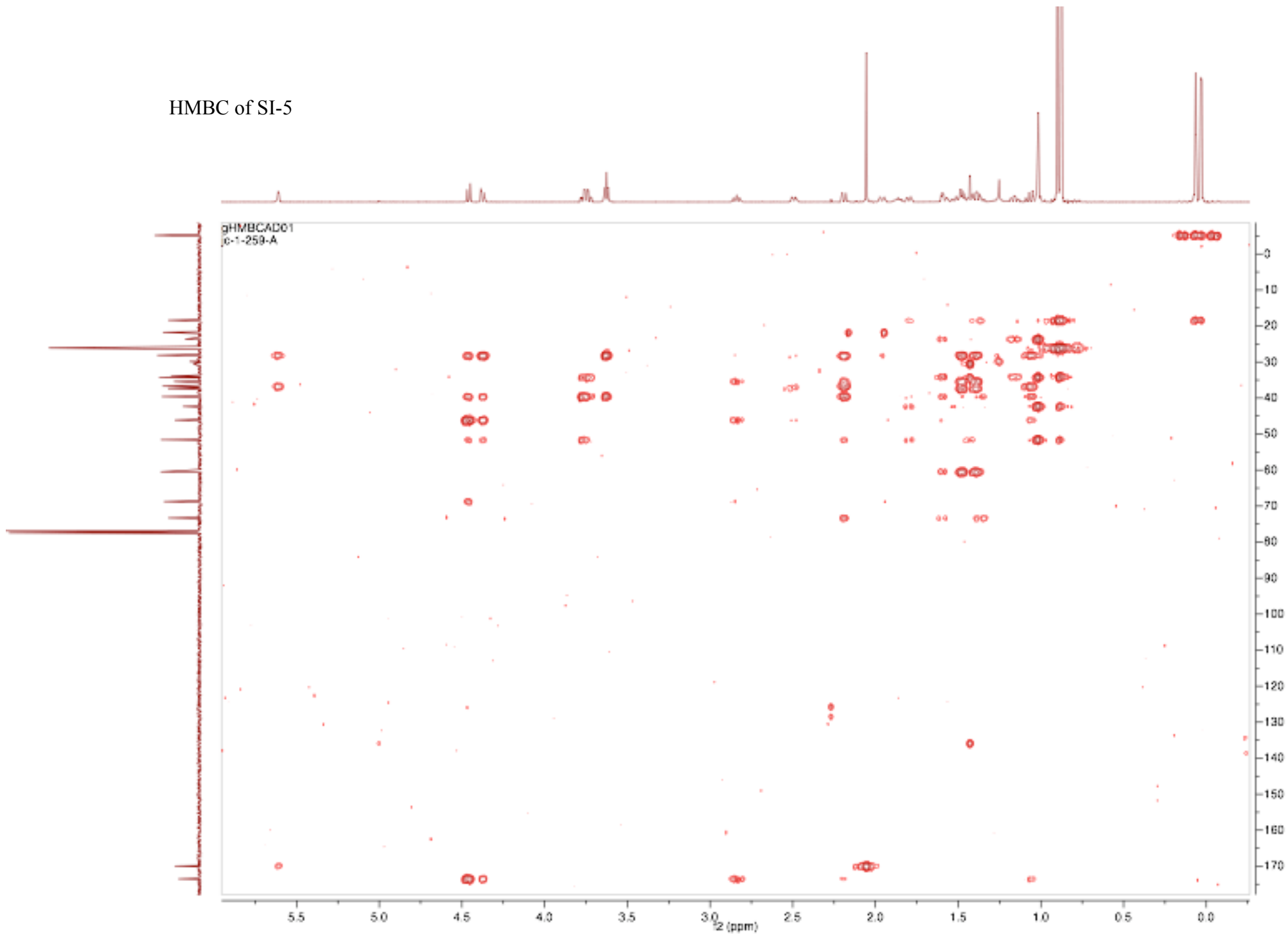
COSY of SI-5



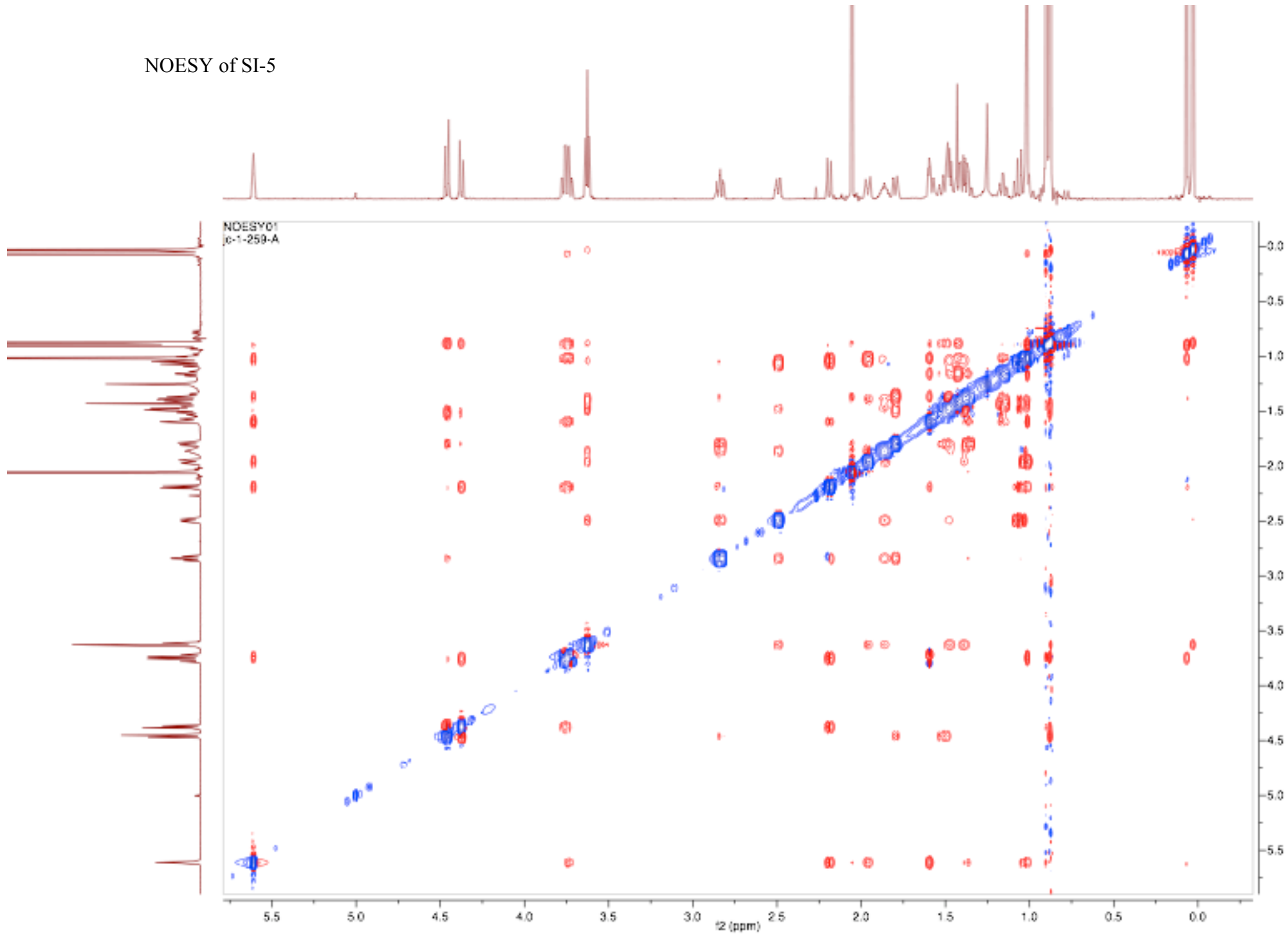
# HSQC of SI-5



HMBC of SI-5



NOESY of SI-5



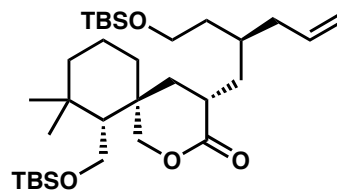
jtsy-vi-105 hi spot

Sample Name:  
jtsy-vi-105hi  
Data Collected on:  
indy.caltech.edu-inova500  
Archive directory:  
/home/yeomanjo/vnmrsys/data  
Sample directory:  
jtsy-vi-105hi  
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Jun 6 2011

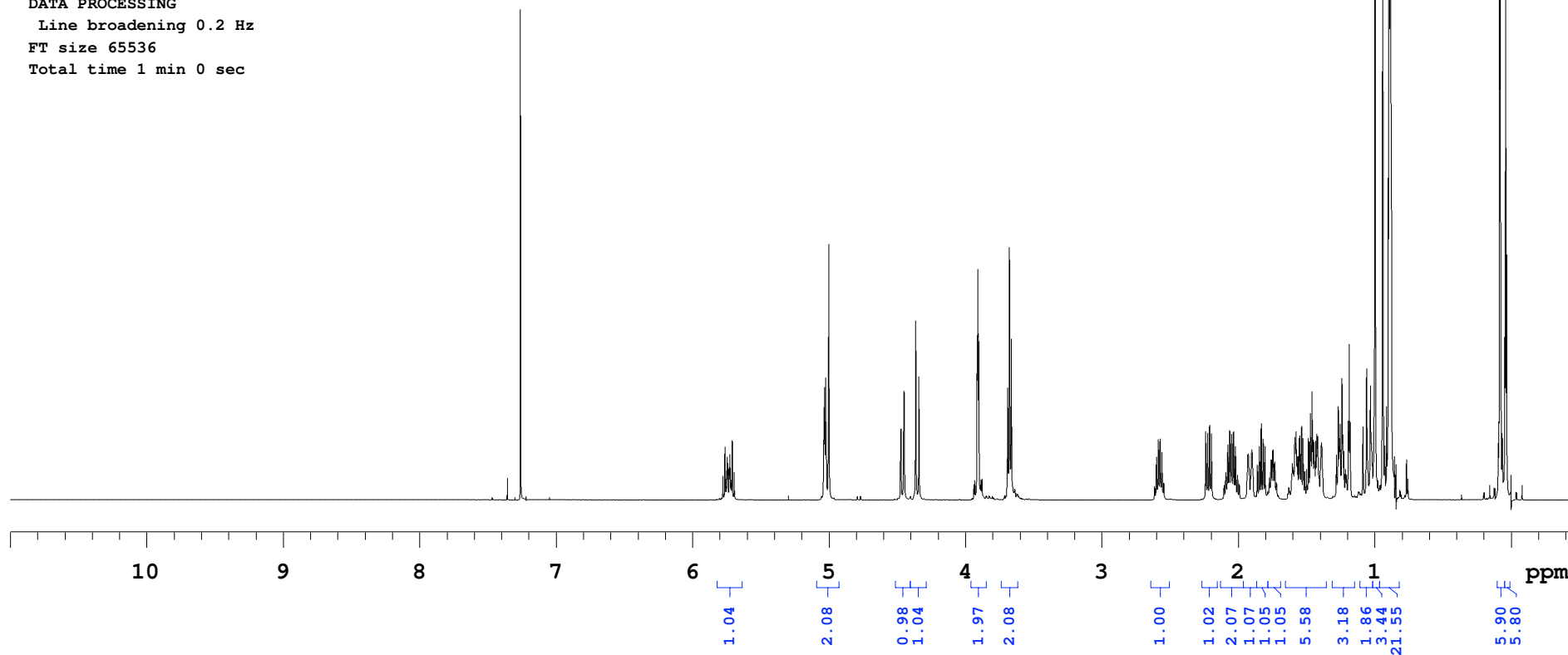
Temp. 25.0 C / 298.1 K  
Sample #12, Operator: yeomanjo

Relax. delay 5.000 sec  
Pulse 45.0 degrees  
Acq. time 2.500 sec  
Width 8000.0 Hz  
8 repetitions  
OBSERVE H1, 499.7290203 MHz  
DATA PROCESSING  
Line broadening 0.2 Hz  
FT size 65536  
Total time 1 min 0 sec



27

*less polar  
diastereomer*



jtsy-vi-105 hi spot

Sample Name:

jtsy-vi-105hi

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/yeomanjo/vnmrsys/data

Sample directory:

jtsy-vi-105hi

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jun 6 2011

Temp. 25.0 C / 298.1 K

Sample #12, Operator: yeomanjo

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1000 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

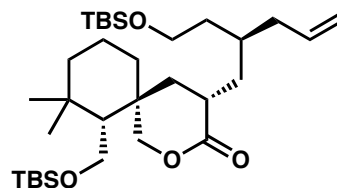
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

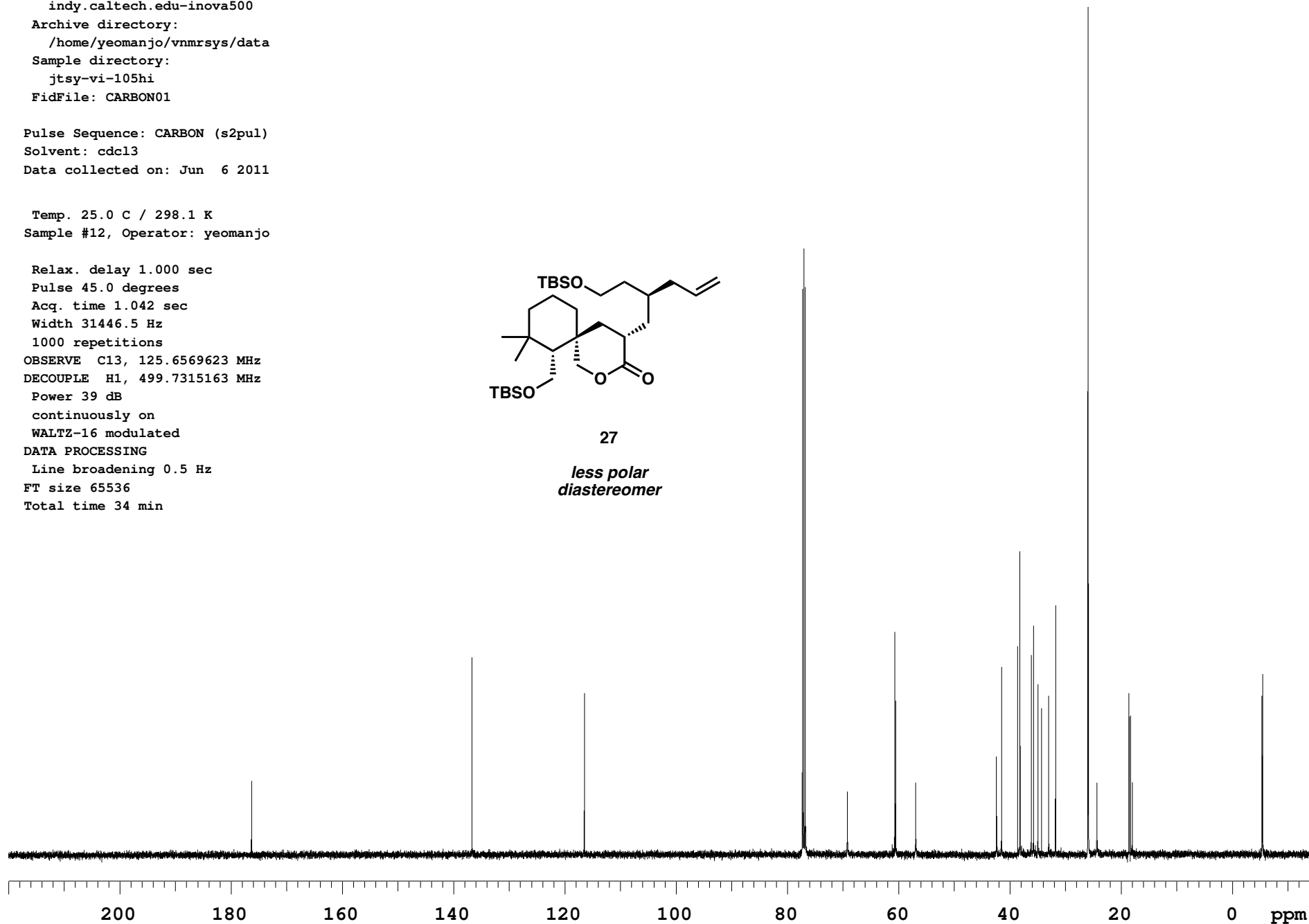
FT size 65536

Total time 34 min



27

*less polar  
diastereomer*

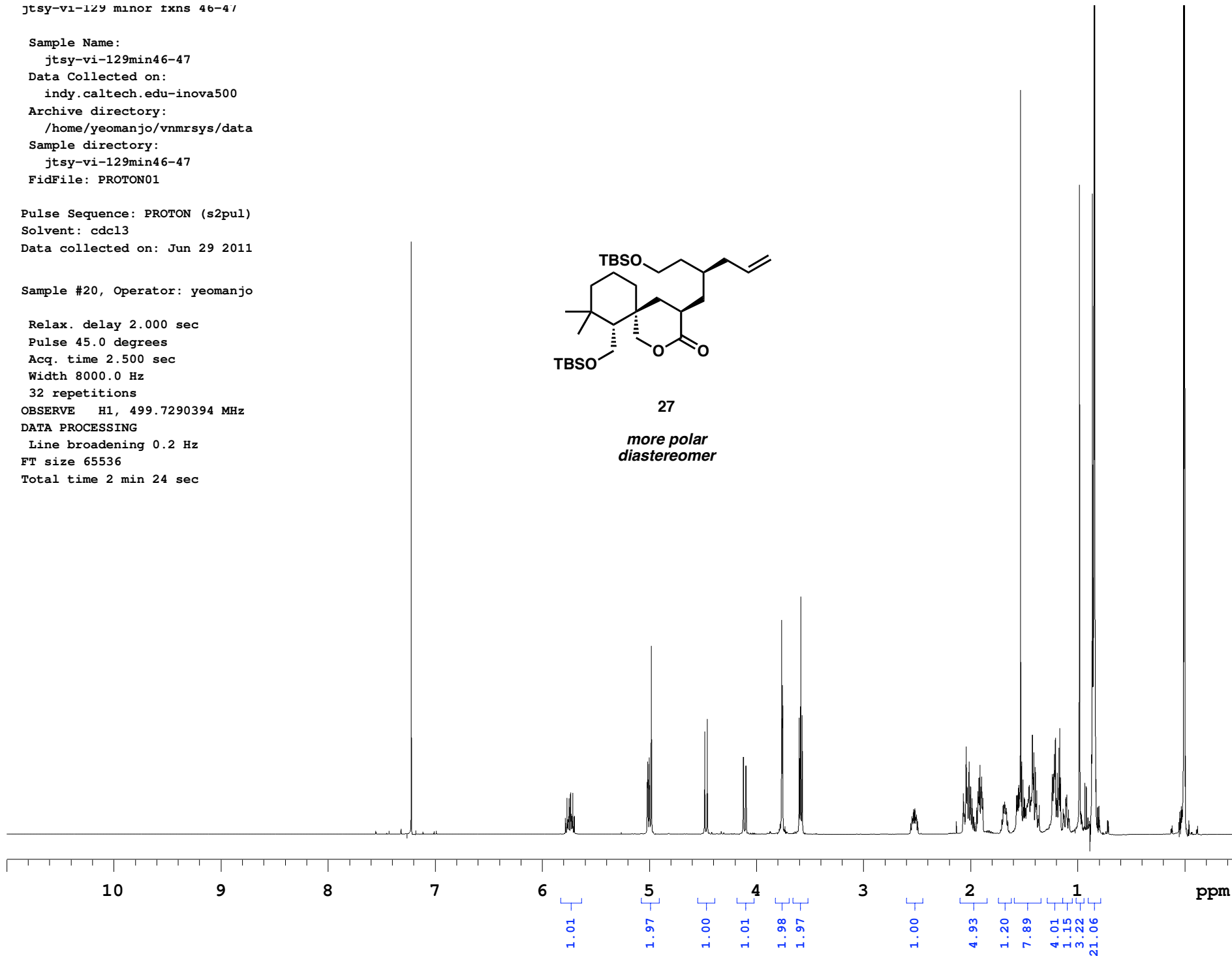
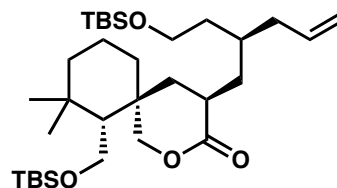


Sample Name:  
jtsy-vi-129min46-47  
Data Collected on:  
indy.caltech.edu-inova500  
Archive directory:  
/home/yeomanjo/vnmrsys/data  
Sample directory:  
jtsy-vi-129min46-47  
FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)  
Solvent: cdcl3  
Data collected on: Jun 29 2011

Sample #20, Operator: yeomanjo

Relax. delay 2.000 sec  
Pulse 45.0 degrees  
Acq. time 2.500 sec  
Width 8000.0 Hz  
32 repetitions  
OBSERVE H1, 499.7290394 MHz  
DATA PROCESSING  
Line broadening 0.2 Hz  
FT size 65536  
Total time 2 min 24 sec





jtsy-vi-129 minor rxns 46-47

Sample Name:

jtsy-vi-129min46-47

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/yeomanjo/vnmrsys/data

Sample directory:

jtsy-vi-129min46-47

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jun 29 2011

Sample #18, Operator: yeomanjo

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

3000 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

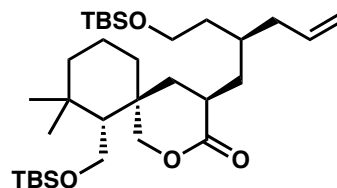
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

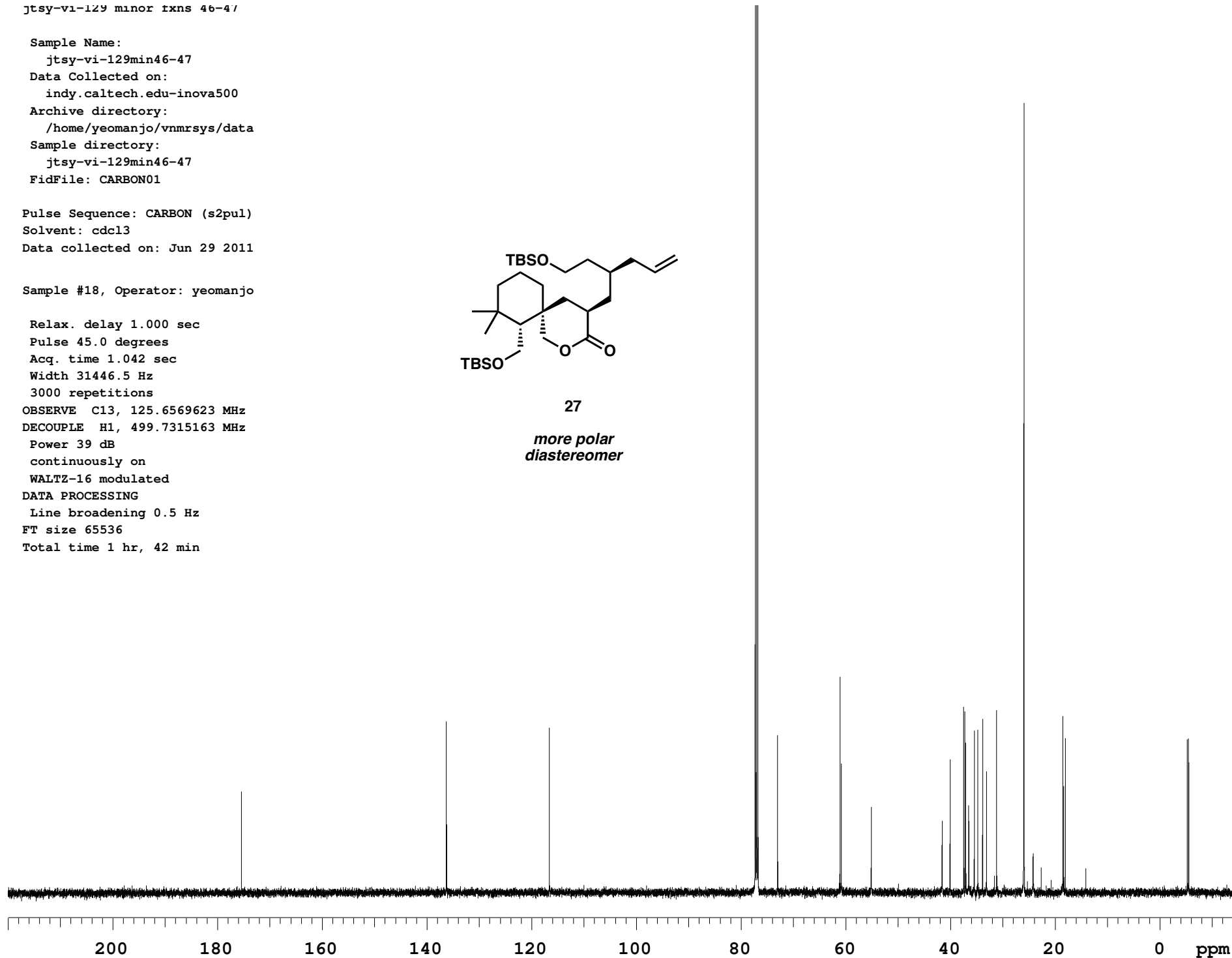
FT size 65536

Total time 1 hr, 42 min



27

*more polar  
diastereomer*



jc-1-288-A

Sample Name:

jc-1-288-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-288-A

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 16 2011

Temp. 25.0 C / 298.1 K

Sample #46, Operator: jcha

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

64 repetitions

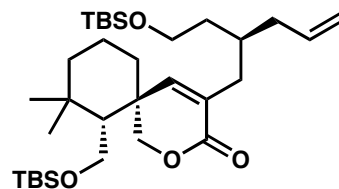
OBSERVE H1, 499.7290200 MHz

DATA PROCESSING

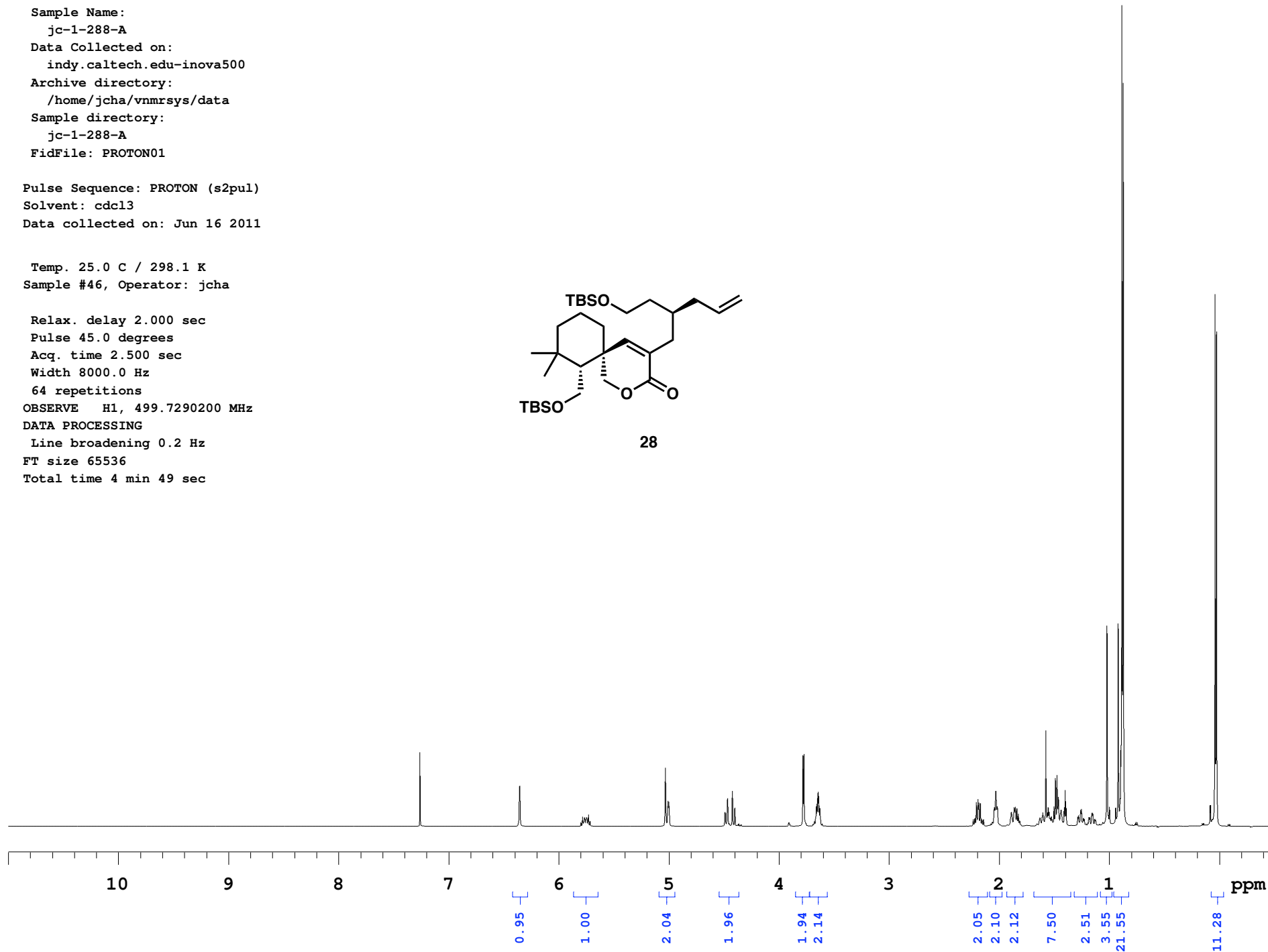
Line broadening 0.2 Hz

FT size 65536

Total time 4 min 49 sec



28



jc-1-288-A

Sample Name:

jc-1-288-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-288-A

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jun 16 2011

Temp. 25.0 C / 298.1 K

Sample #46, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1000 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

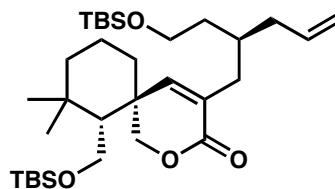
WALTZ-16 modulated

DATA PROCESSING

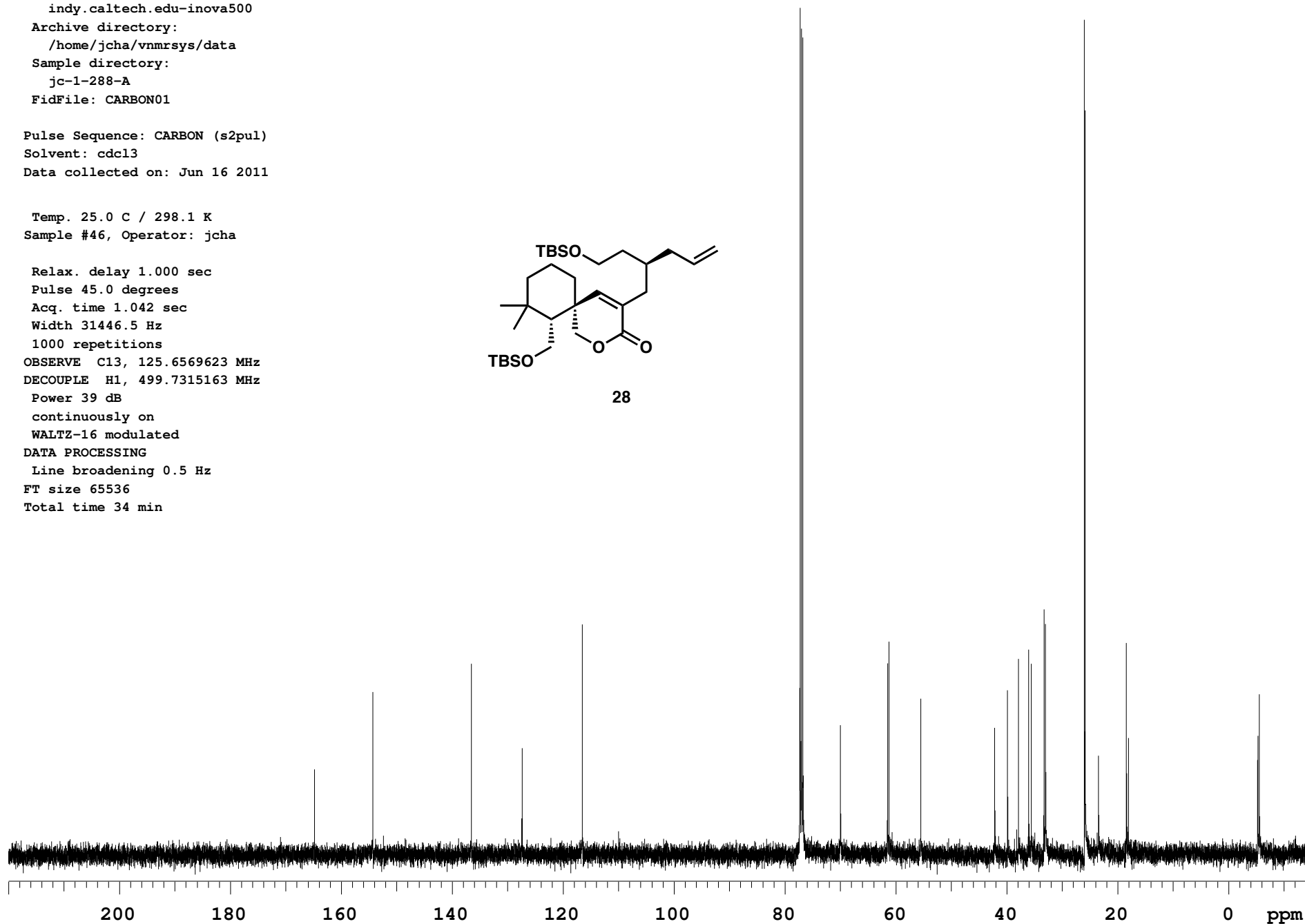
Line broadening 0.5 Hz

FT size 65536

Total time 34 min



28



jc-1-272-A-char

Sample Name:

jc-1-272-A-char

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-272-A-char

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 2 2011

Temp. 25.0 C / 298.1 K

Sample #2, Operator: jcha

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

64 repetitions

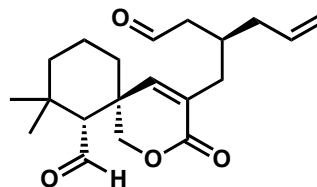
OBSERVE H1, 499.7290203 MHz

DATA PROCESSING

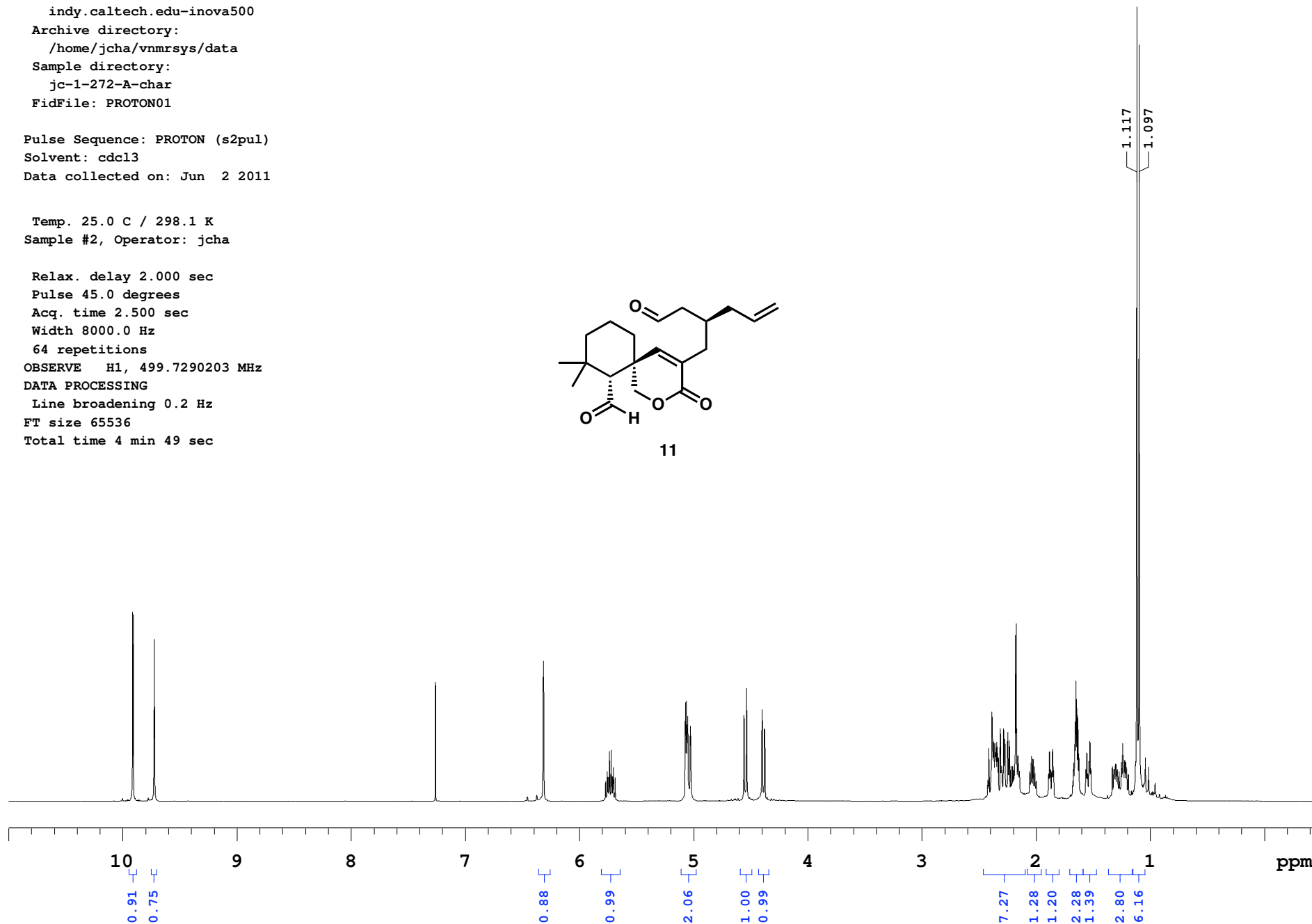
Line broadening 0.2 Hz

FT size 65536

Total time 4 min 49 sec



11



jc-1-272-A-char

Sample Name:

jc-1-272-A-char

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-272-A-char

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jun 2 2011

Temp. 25.0 C / 298.1 K

Sample #2, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

512 repetitions

OBSERVE C13, 125.6569642 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

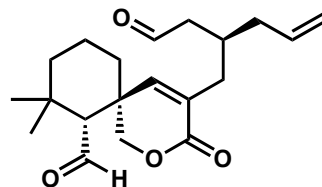
WALTZ-16 modulated

DATA PROCESSING

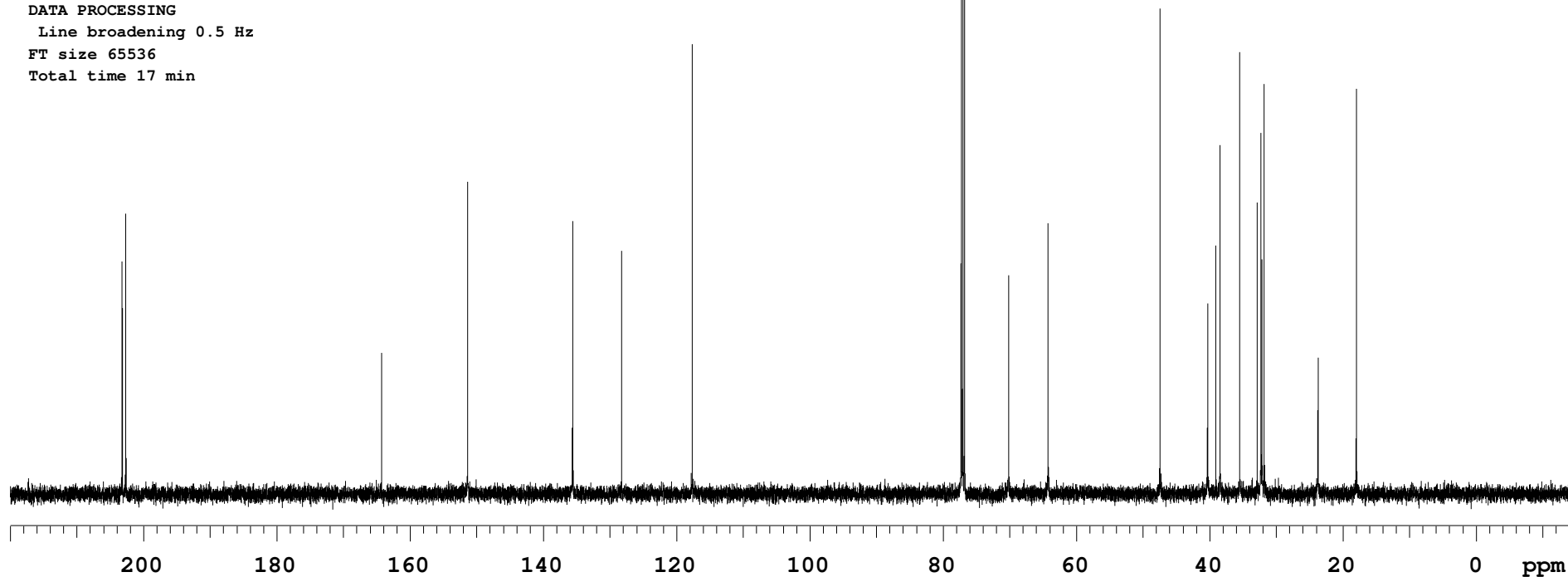
Line broadening 0.5 Hz

FT size 65536

Total time 17 min



11



jtsy-vi-163pHPLC

Sample Name:

jtsy-vi-163pHPLC

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/yeomanjo/vnmrsys/data

Sample directory:

jtsy-vi-163pHPLC

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jul 1 2011

Sample #14, Operator: yeomanjo

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

32 repetitions

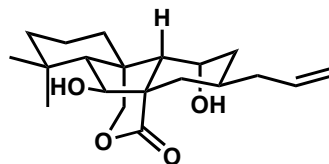
OBSERVE H1, 499.7290166 MHz

DATA PROCESSING

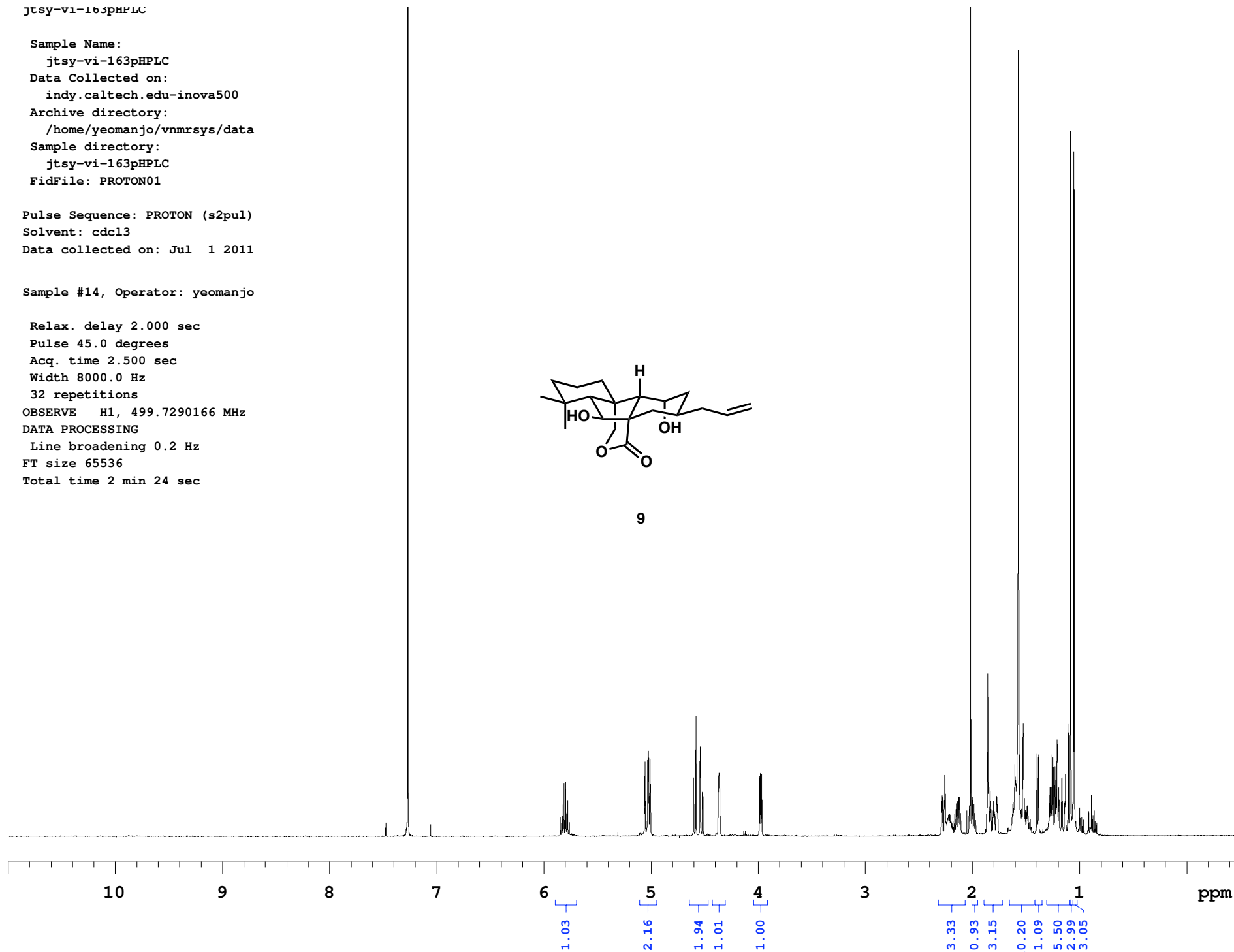
Line broadening 0.2 Hz

FT size 65536

Total time 2 min 24 sec



9



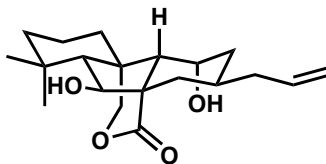
jtsy-vi-163

Sample Name:  
jtsy-vi-163pHPLC  
Data Collected on:  
indy.caltech.edu-inova500  
Archive directory:  
/home/yeomanjo/vnmrsys/data  
Sample directory:  
jtsy-vi-163pHPLC  
FidFile: CARBON01

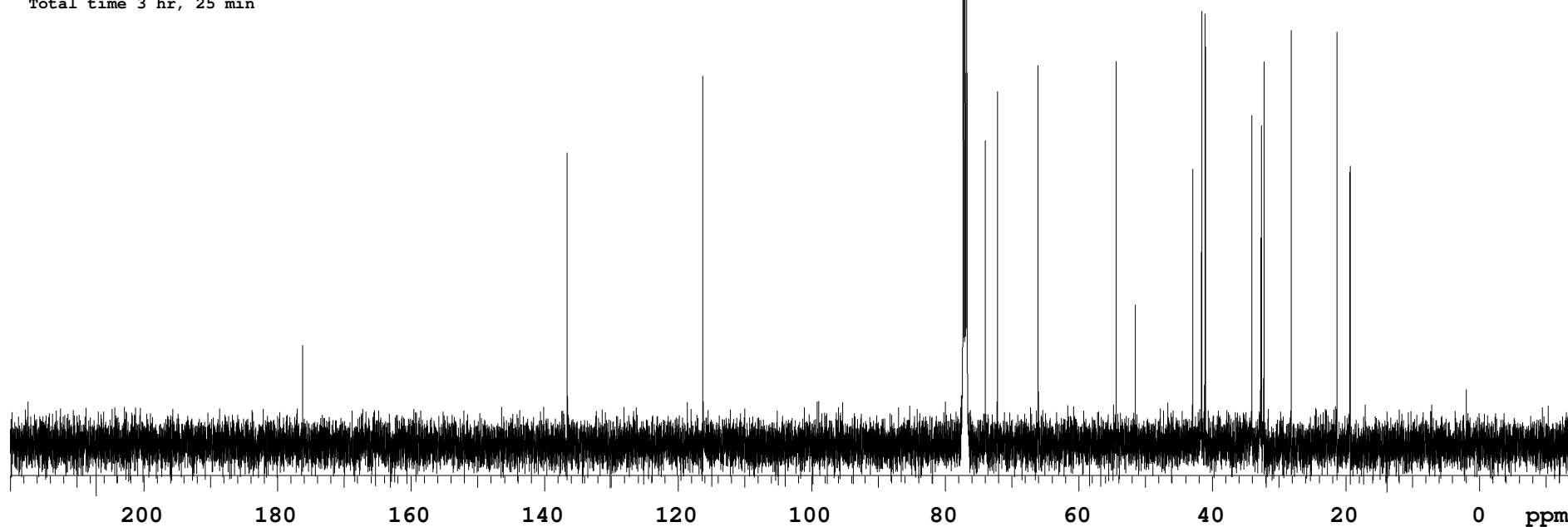
Pulse Sequence: CARBON (s2pul)  
Solvent: cdcl3  
Data collected on: Jul 1 2011

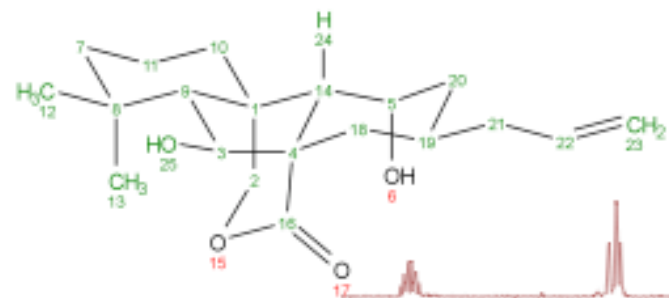
Sample #14, Operator: yeomanjo

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 1.042 sec  
Width 31446.5 Hz  
6000 repetitions  
OBSERVE C13, 125.6569623 MHz  
DECOUPLE H1, 499.7315163 MHz  
Power 39 dB  
continuously on  
WALTZ-16 modulated  
DATA PROCESSING  
Line broadening 0.5 Hz  
FT size 65536  
Total time 3 hr, 25 min

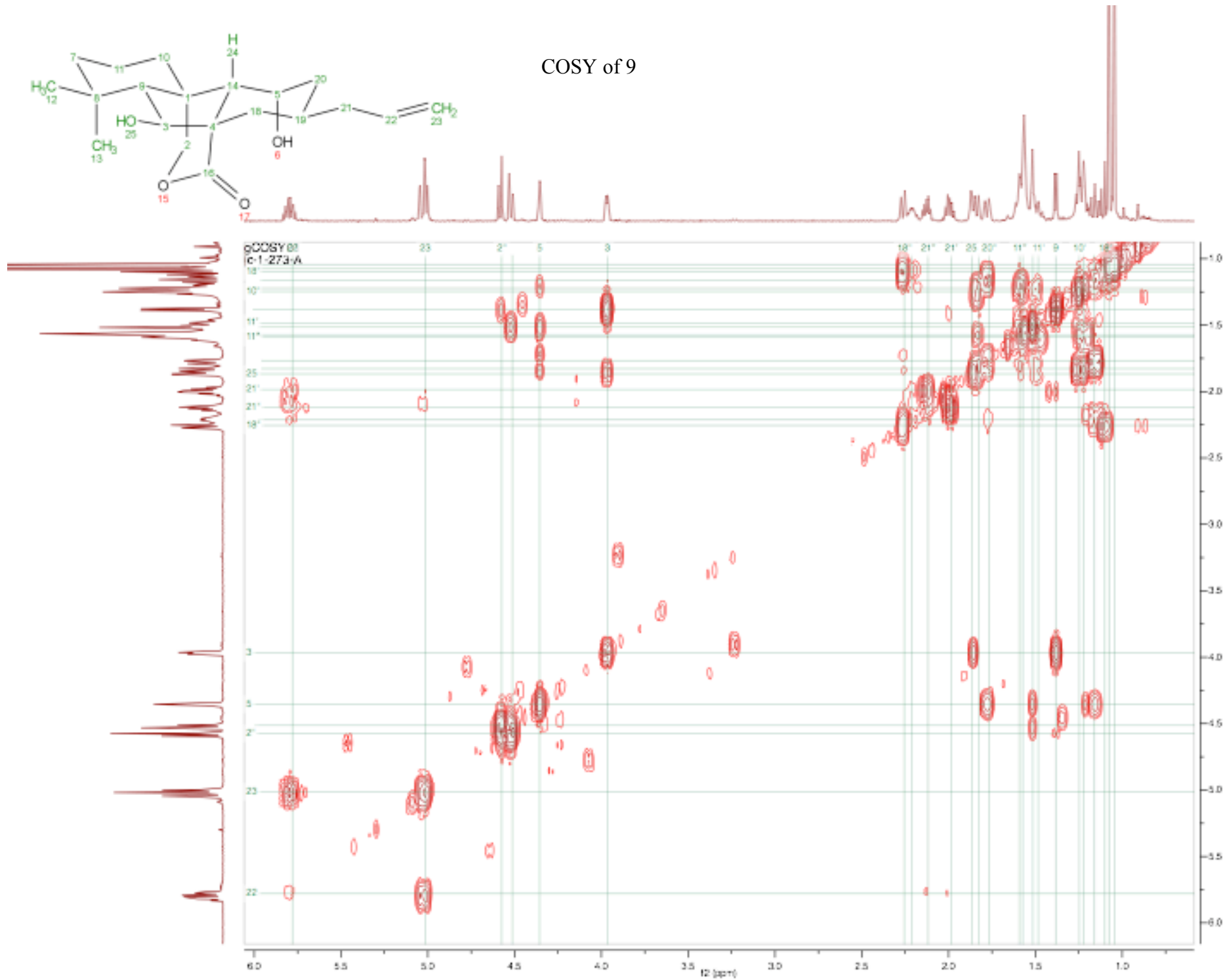


9

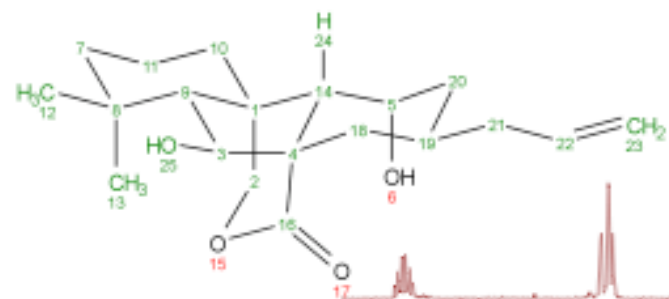




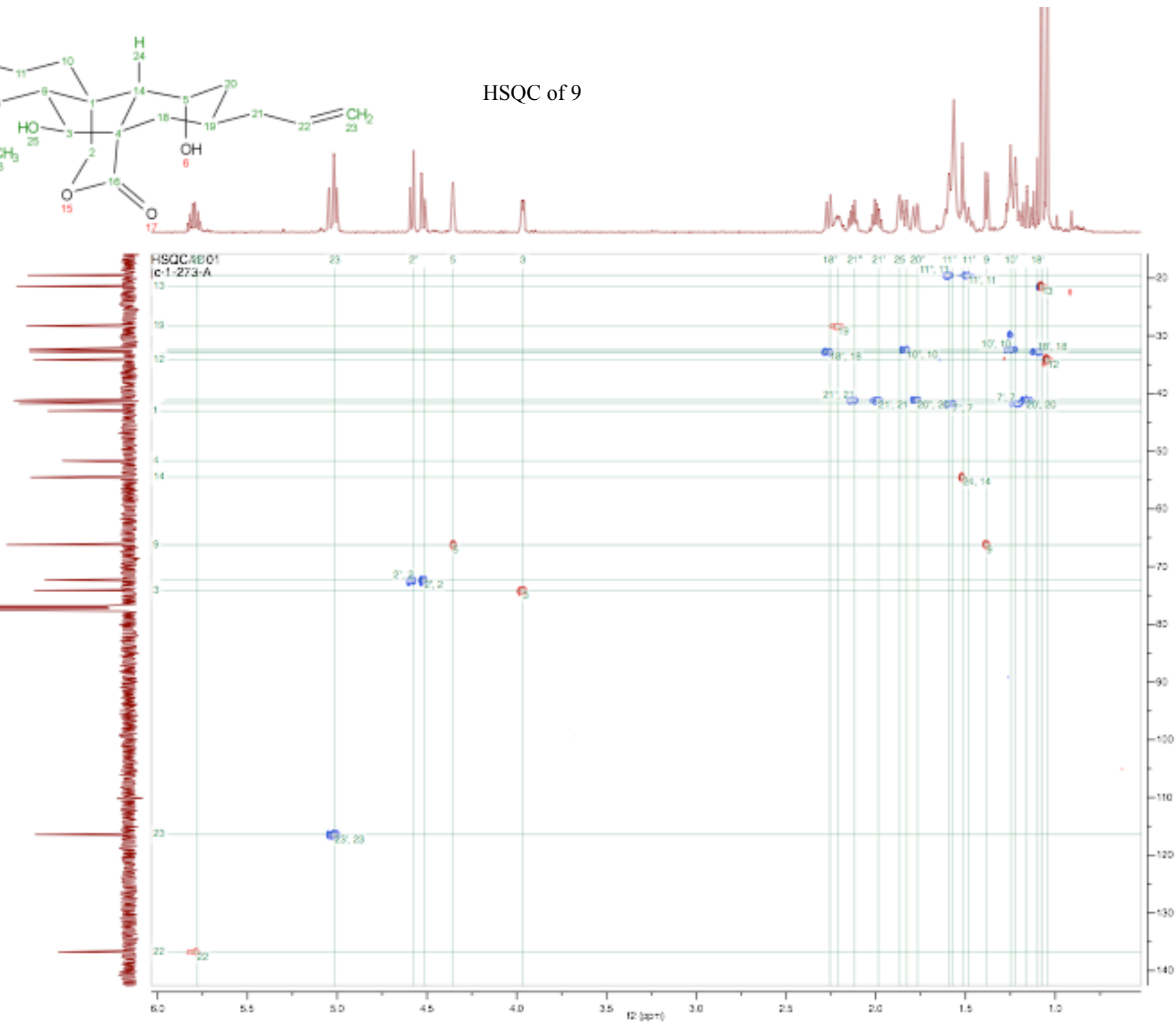
COSY of 9

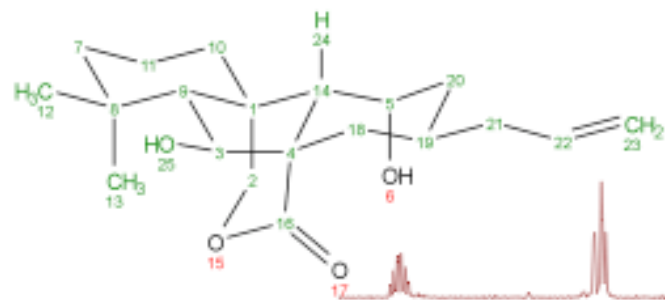




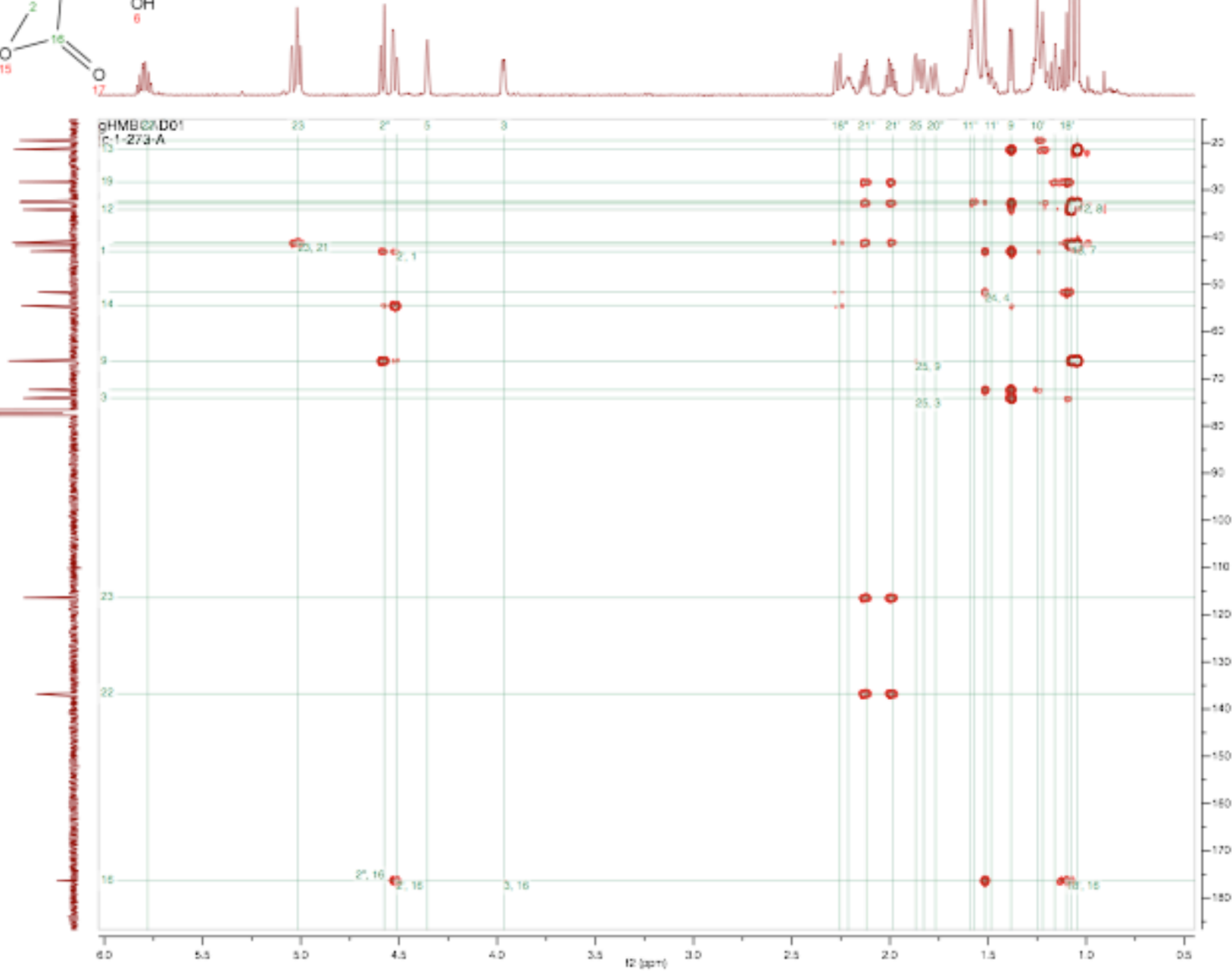


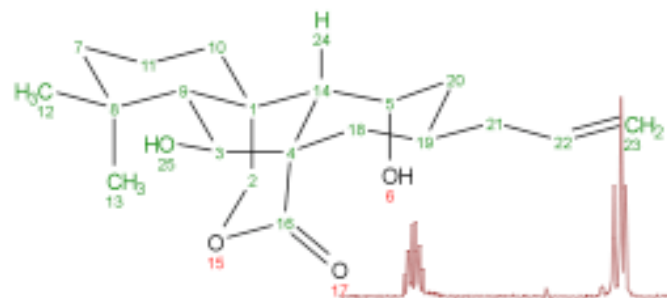
HSQC of 9



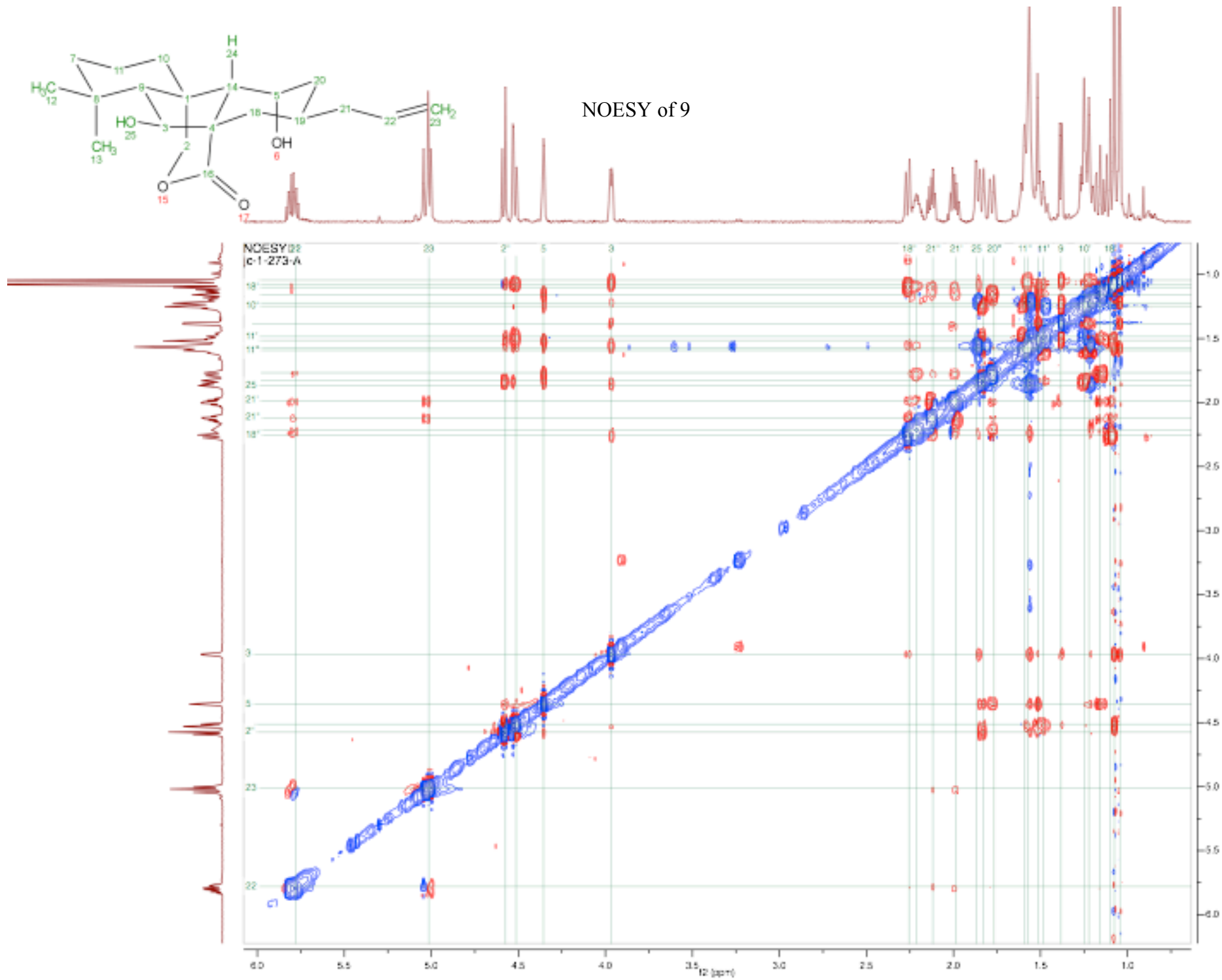


HMBC of 9





NOESY of 9



jc-1-274-A

Sample Name:

jc-1-274-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-274-A

FidFile: current

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jul 20 2011

Sample #2, Operator: jcha

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

32 repetitions

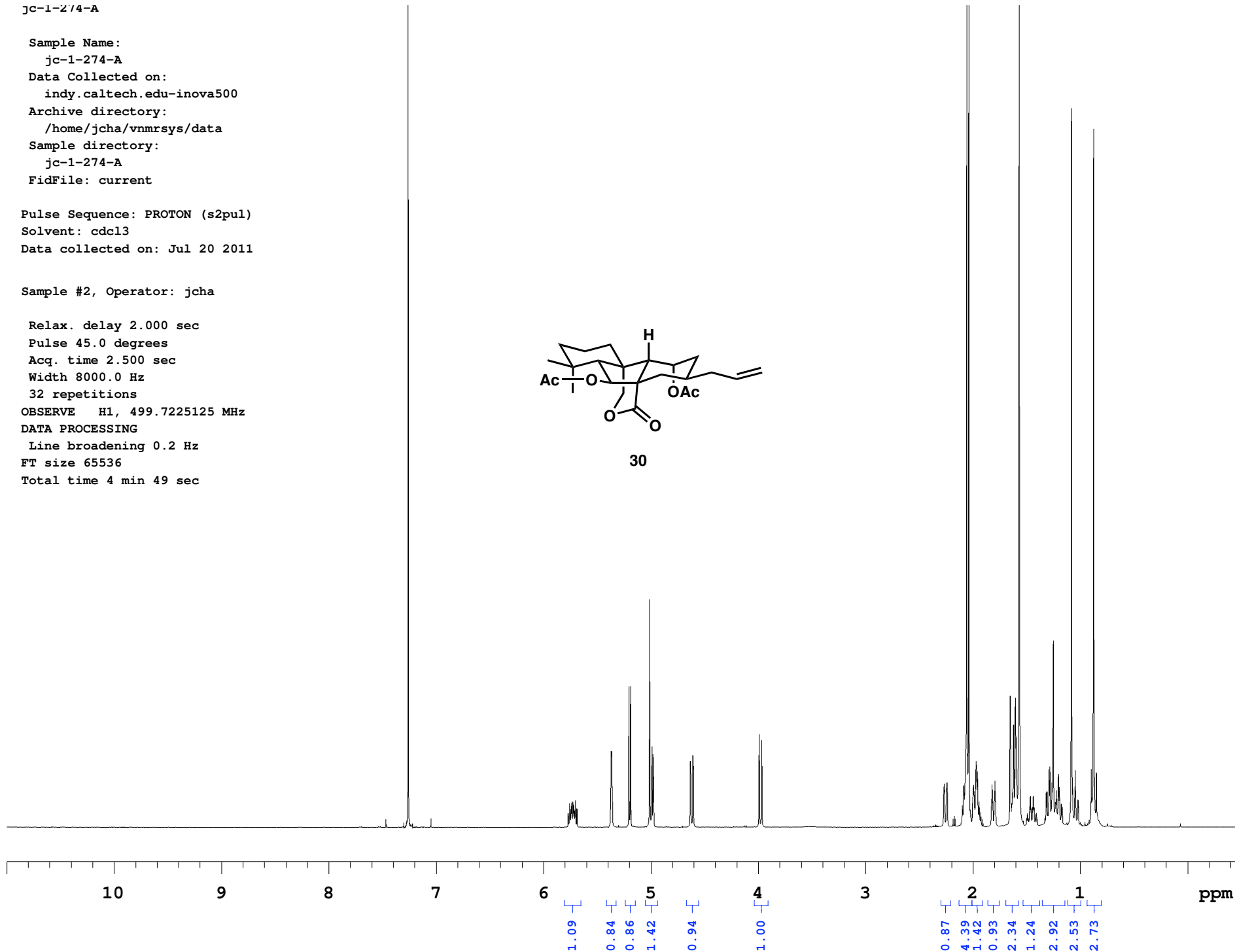
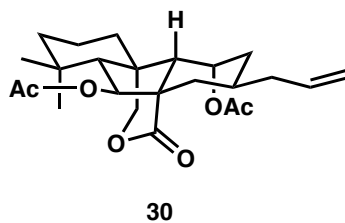
OBSERVE H1, 499.7225125 MHz

DATA PROCESSING

Line broadening 0.2 Hz

FT size 65536

Total time 4 min 49 sec



jc-1-2/4-A

Sample Name:

jc-1-274-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-274-A

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jul 18 2011

Sample #46, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

1725 repetitions

OBSERVE C13, 125.6553262 MHz

DECOUPLE H1, 499.7250019 MHz

Power 39 dB

continuously on

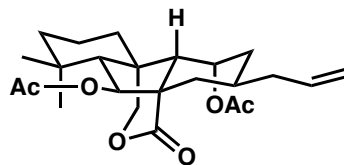
WALTZ-16 modulated

DATA PROCESSING

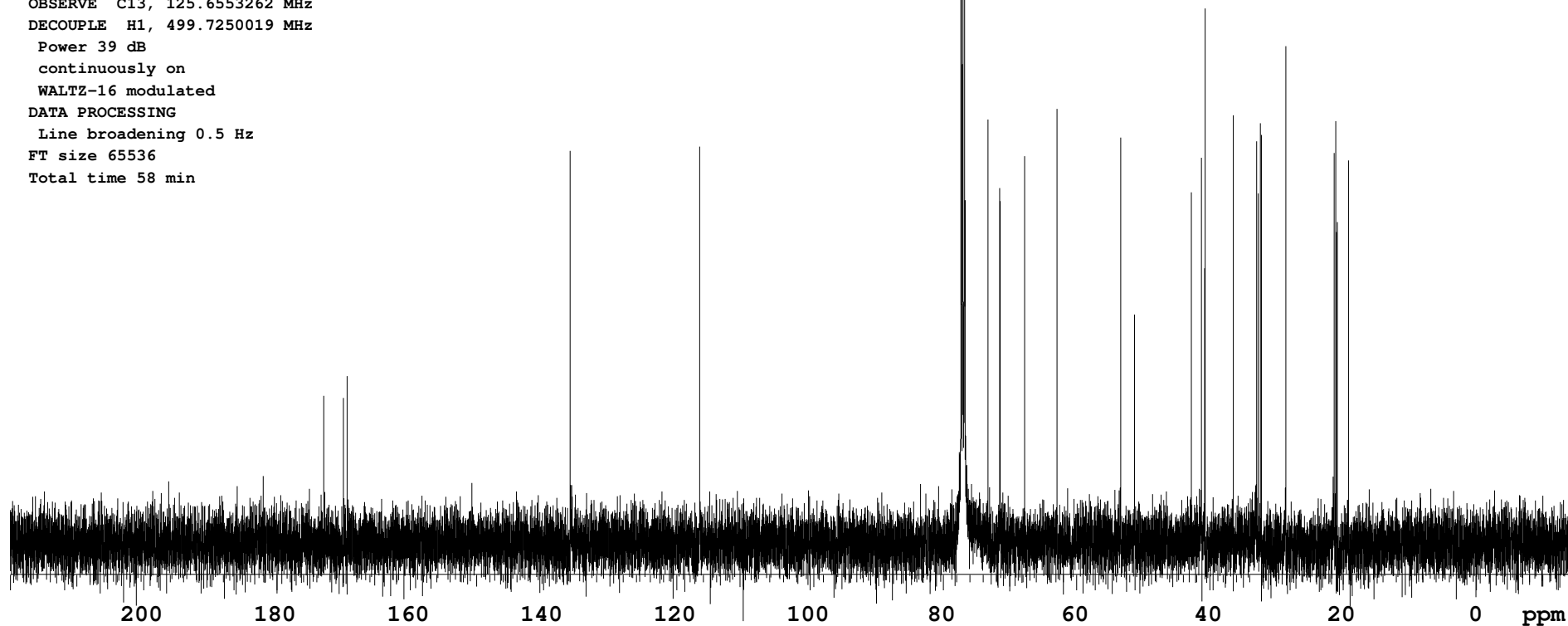
Line broadening 0.5 Hz

FT size 65536

Total time 58 min



30



jc-1-2/4-B

Sample Name:

jc-1-274-B

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-274-B

FidFile: PROTON03

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jul 26 2011

Sample #1, Operator: jcha

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

64 repetitions

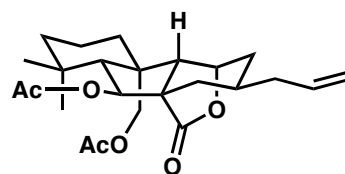
OBSERVE H1, 499.7225125 MHz

DATA PROCESSING

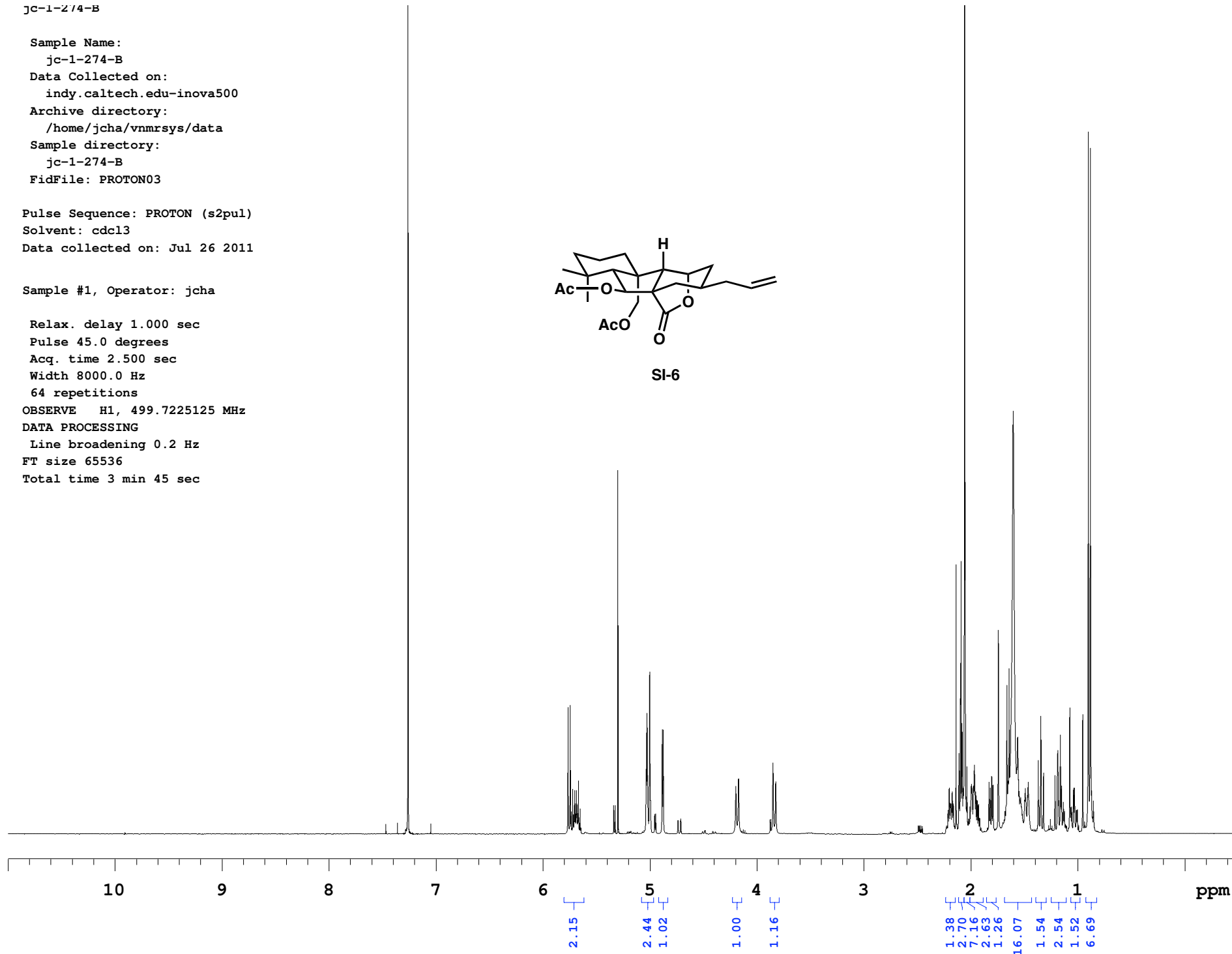
Line broadening 0.2 Hz

FT size 65536

Total time 3 min 45 sec



SI-6



jc-1-274-B

Sample Name:

jc-1-274-B

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

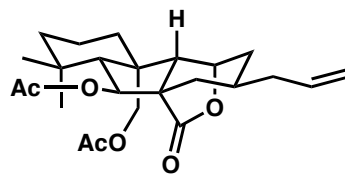
jc-1-274-B

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jul 26 2011



SI-6

Sample #1, Operator: jcha

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

2250 repetitions

OBSERVE C13, 125.6553271 MHz

DECOUPLE H1, 499.7250019 MHz

Power 39 dB

continuously on

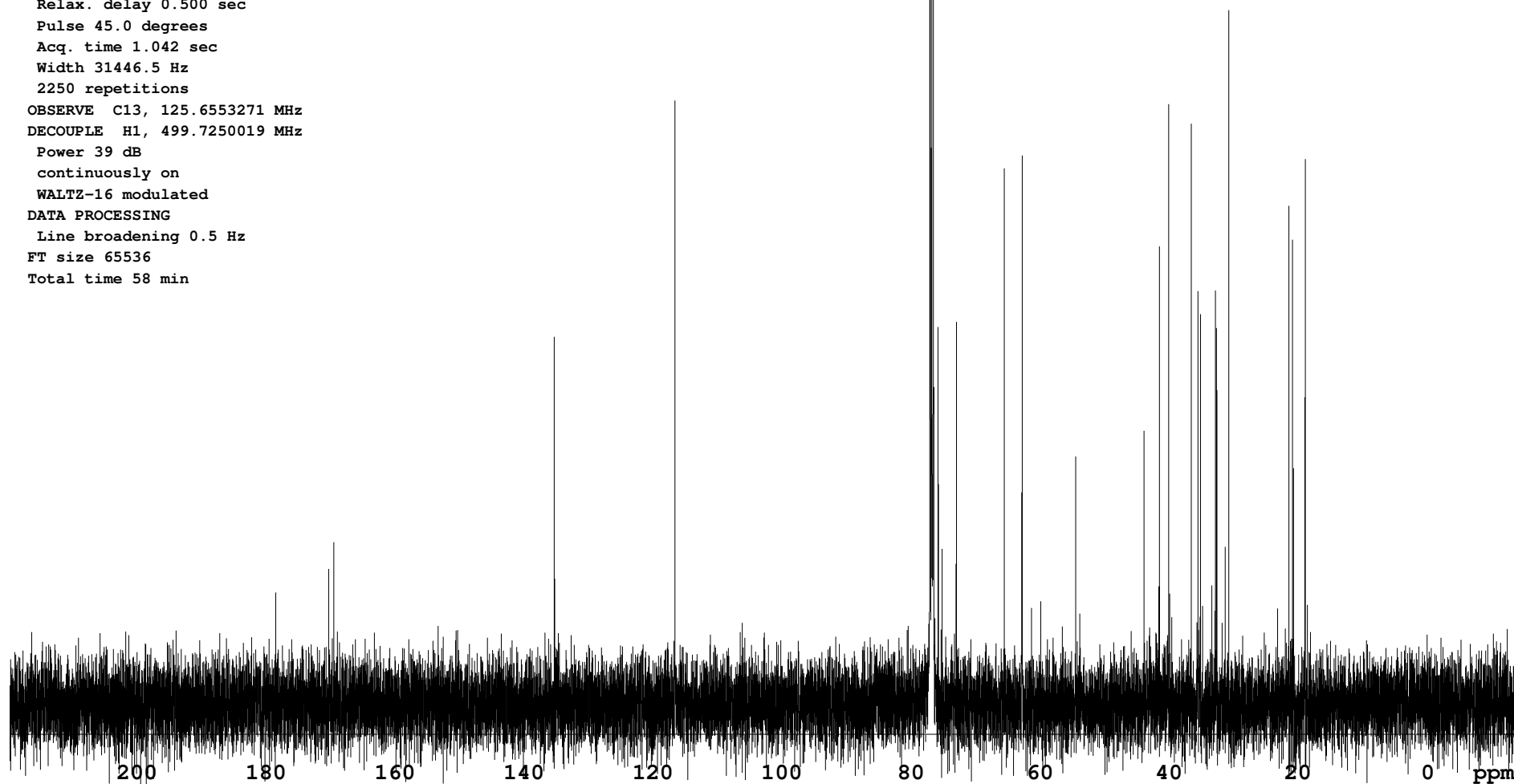
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 65536

Total time 58 min



jc-1-276-A

Sample Name:

jc-1-276-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-276-A

FidFile: PROTON02

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Jun 30 2011

Sample #1, Operator: jcha

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

32 repetitions

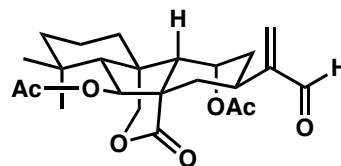
OBSERVE H1, 499.7290200 MHz

DATA PROCESSING

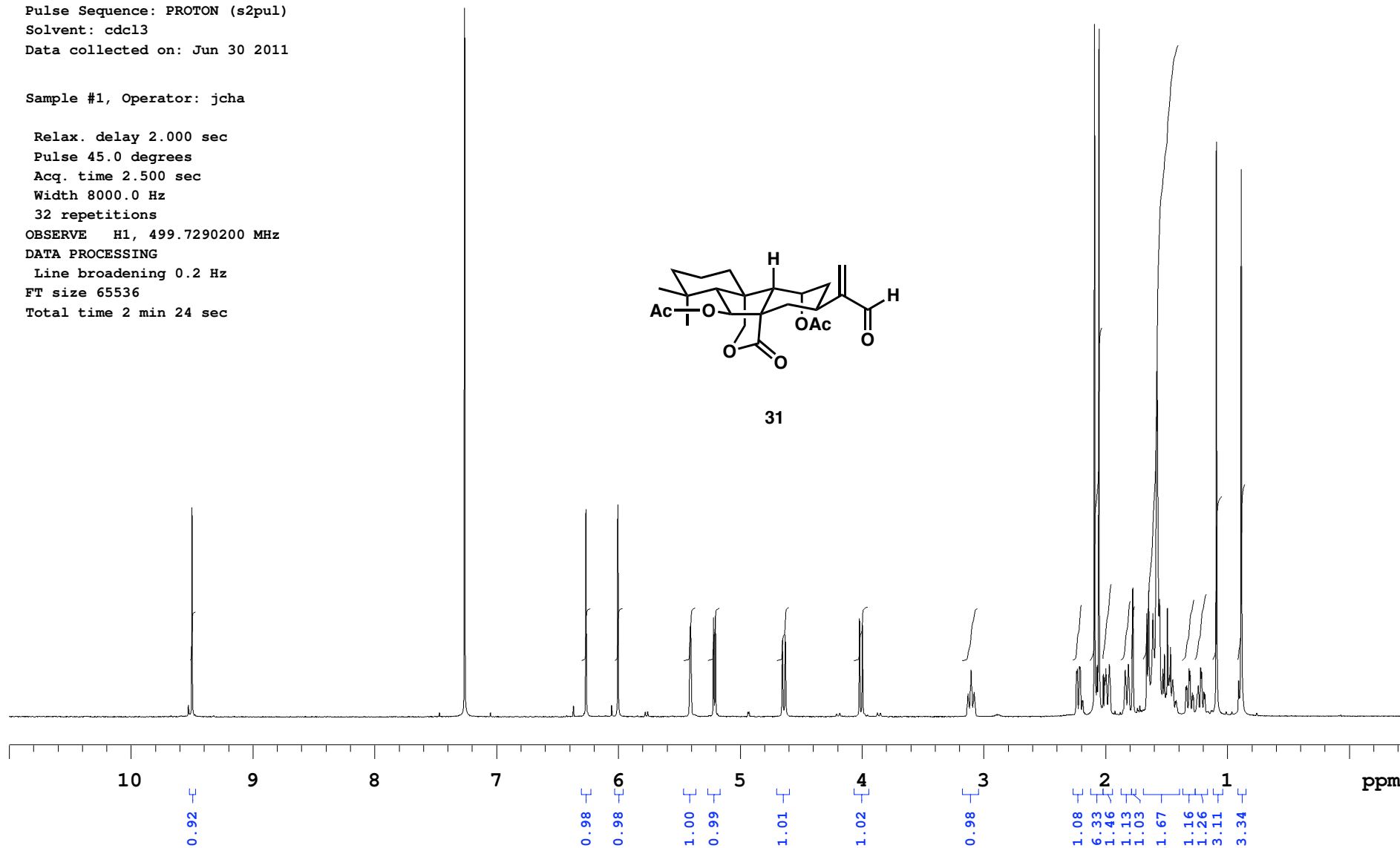
Line broadening 0.2 Hz

FT size 65536

Total time 2 min 24 sec



31





jc-1-276-A

Sample Name:

jc-1-276-A

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-276-A

FidFile: CARBON01

Pulse Sequence: CARBON (s2pul)

Solvent: cdcl3

Data collected on: Jun 30 2011

Sample #1, Operator: jcha

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

9400 repetitions

OBSERVE C13, 125.6569623 MHz

DECOUPLE H1, 499.7315163 MHz

Power 39 dB

continuously on

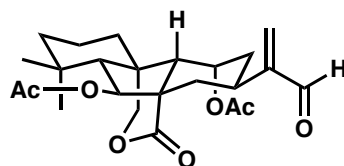
WALTZ-16 modulated

DATA PROCESSING

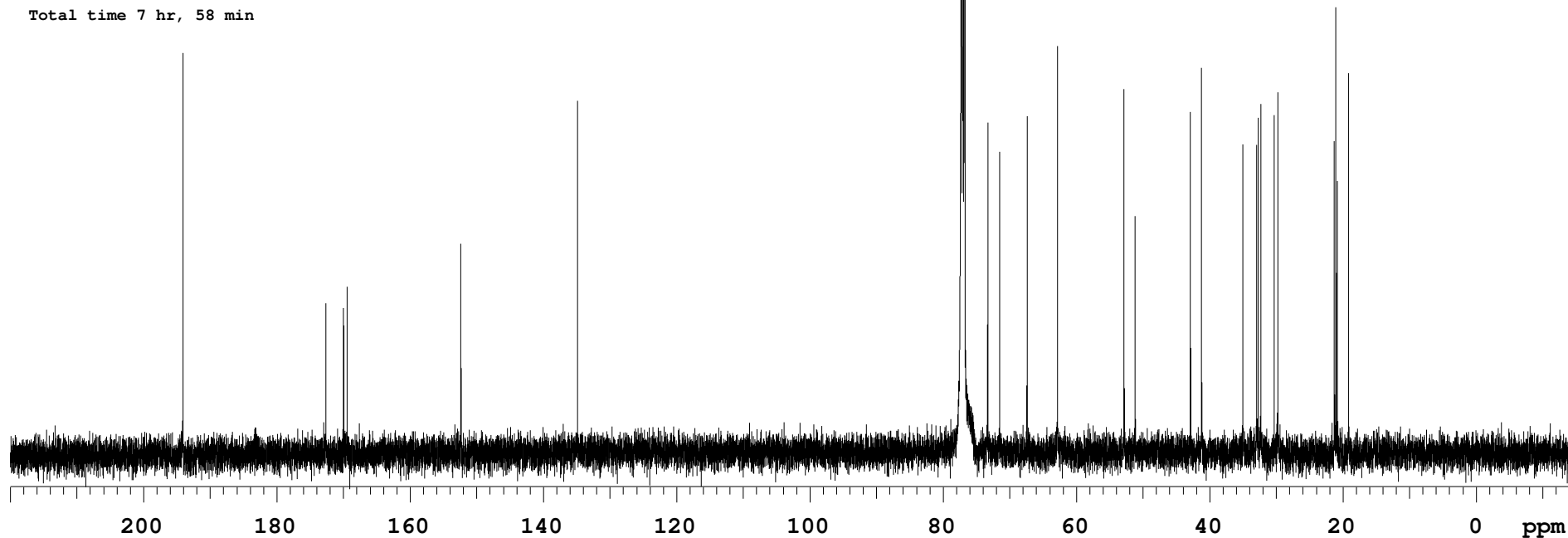
Line broadening 0.5 Hz

FT size 65536

Total time 7 hr, 58 min



31



jc-1-277-pyridine

Sample Name:

jc-1-277-pyridine

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-277-pyridine

FidFile: PROTON01

Pulse Sequence: PROTON (s2pul)

Solvent: pyridine

Data collected on: Aug 2 2011

Sample #46, Operator: jcha

Relax. delay 3.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

128 repetitions

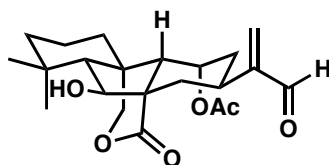
OBSERVE H1, 499.7217632 MHz

DATA PROCESSING

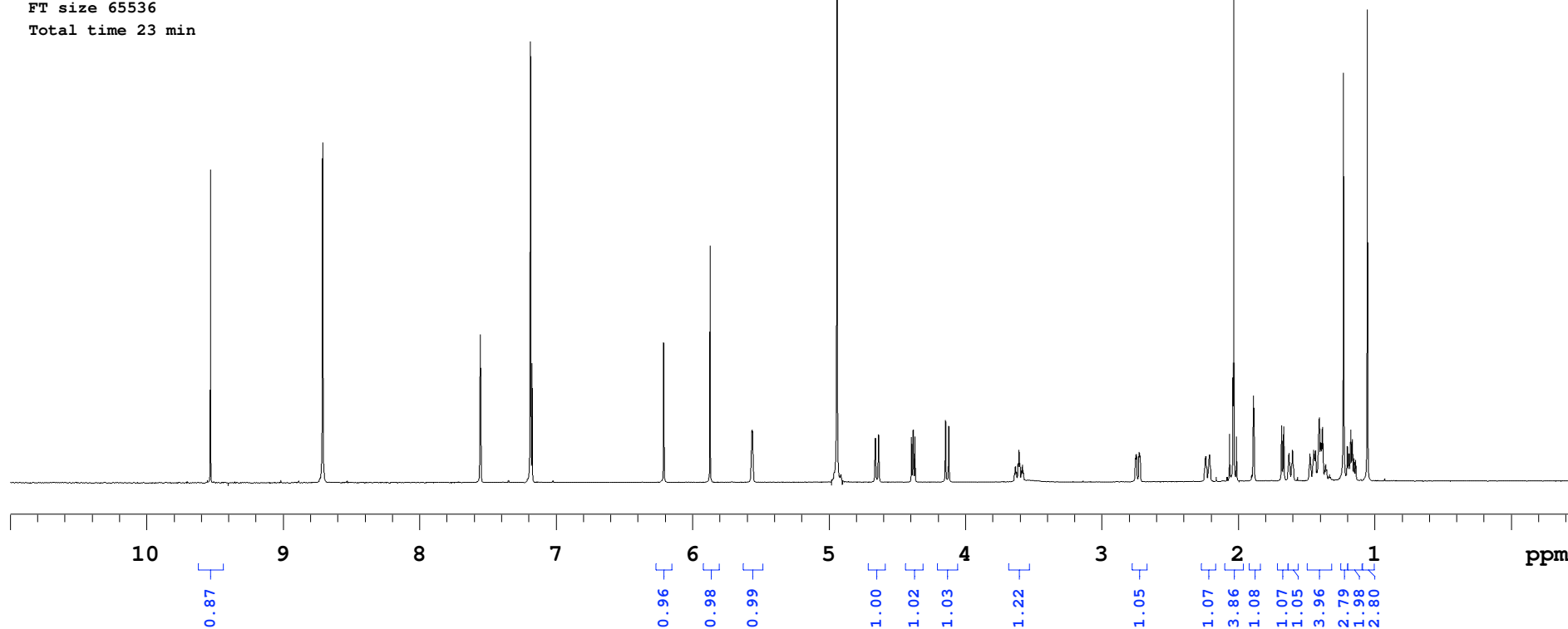
Line broadening 0.2 Hz

FT size 65536

Total time 23 min



*maoecrystal Z (6)*



jc-1-277-pyridine

Sample Name:

jc-1-277-pyridine

Data Collected on:

indy.caltech.edu-inova500

Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-277-pyridine

FidFile: CARBON02

Pulse Sequence: CARBON (s2pul)

Solvent: pyridine

Data collected on: Aug 2 2011

Sample #1, Operator: jcha

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 1.042 sec

Width 31446.5 Hz

15000 repetitions

OBSERVE C13, 125.6552985 MHz

DECOUPLE H1, 499.7248470 MHz

Power 39 dB

continuously on

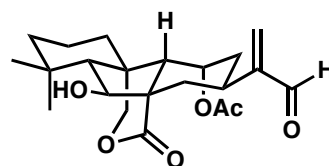
WALTZ-16 modulated

DATA PROCESSING

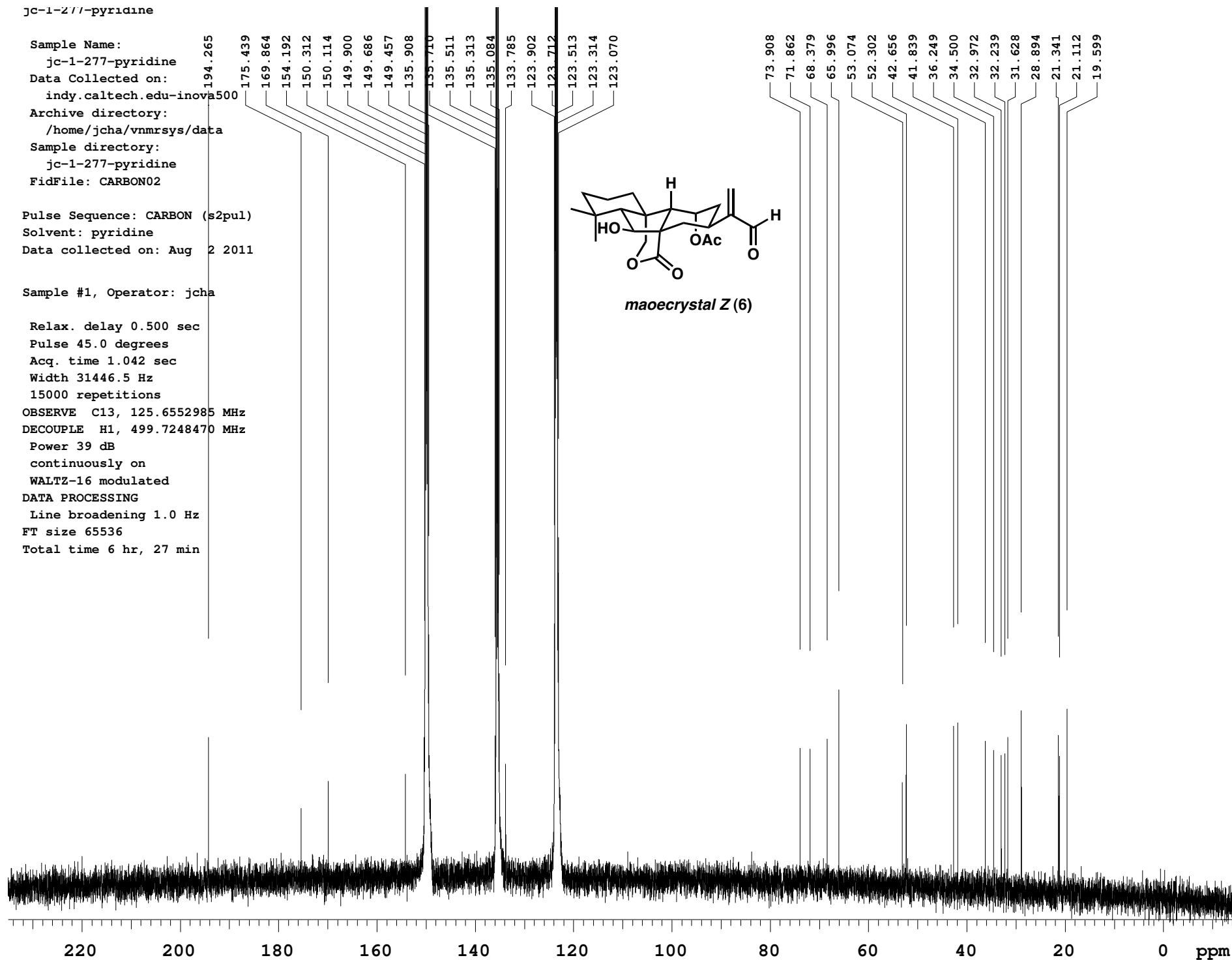
Line broadening 1.0 Hz

FT size 65536

Total time 6 hr, 27 min



maoecrystal Z (6)



jc-1-277-cdcl3

Sample Name:

jc-1-277-cdcl3

Data Collected on:

indy.caltech.edu-inova500

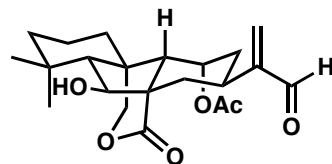
Archive directory:

/home/jcha/vnmrsys/data

Sample directory:

jc-1-277-cdcl3

FidFile: PROTON01



*maoecrystal Z (6)*

Pulse Sequence: PROTON (s2pul)

Solvent: cdcl3

Data collected on: Aug 2 2011

Sample #46, Operator: jcha

Relax. delay 3.000 sec

Pulse 45.0 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

128 repetitions

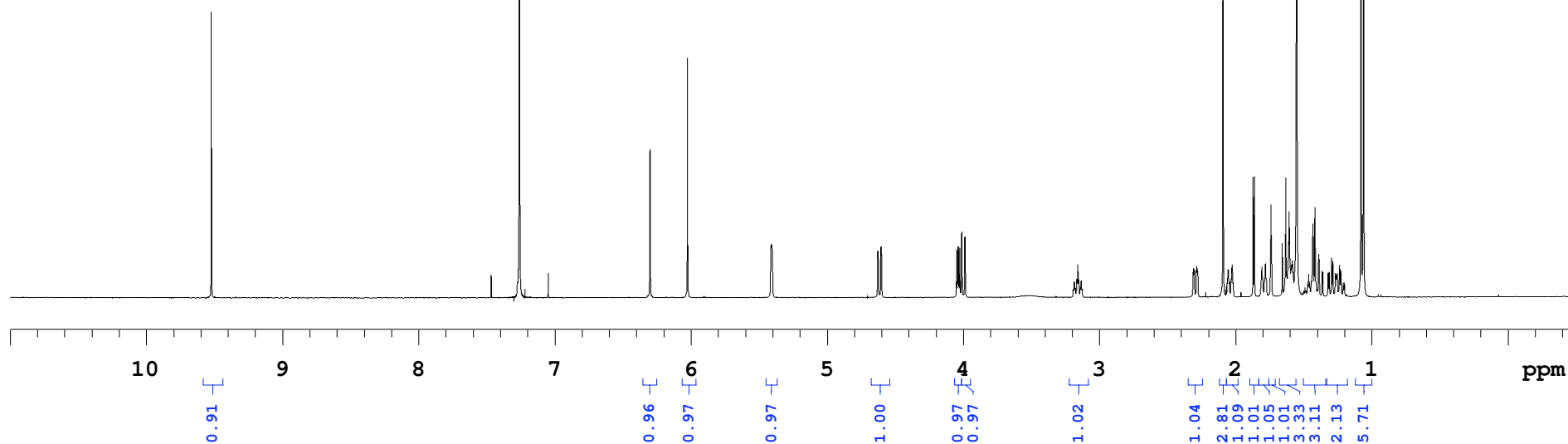
OBSERVE H1, 499.7225128 MHz

DATA PROCESSING

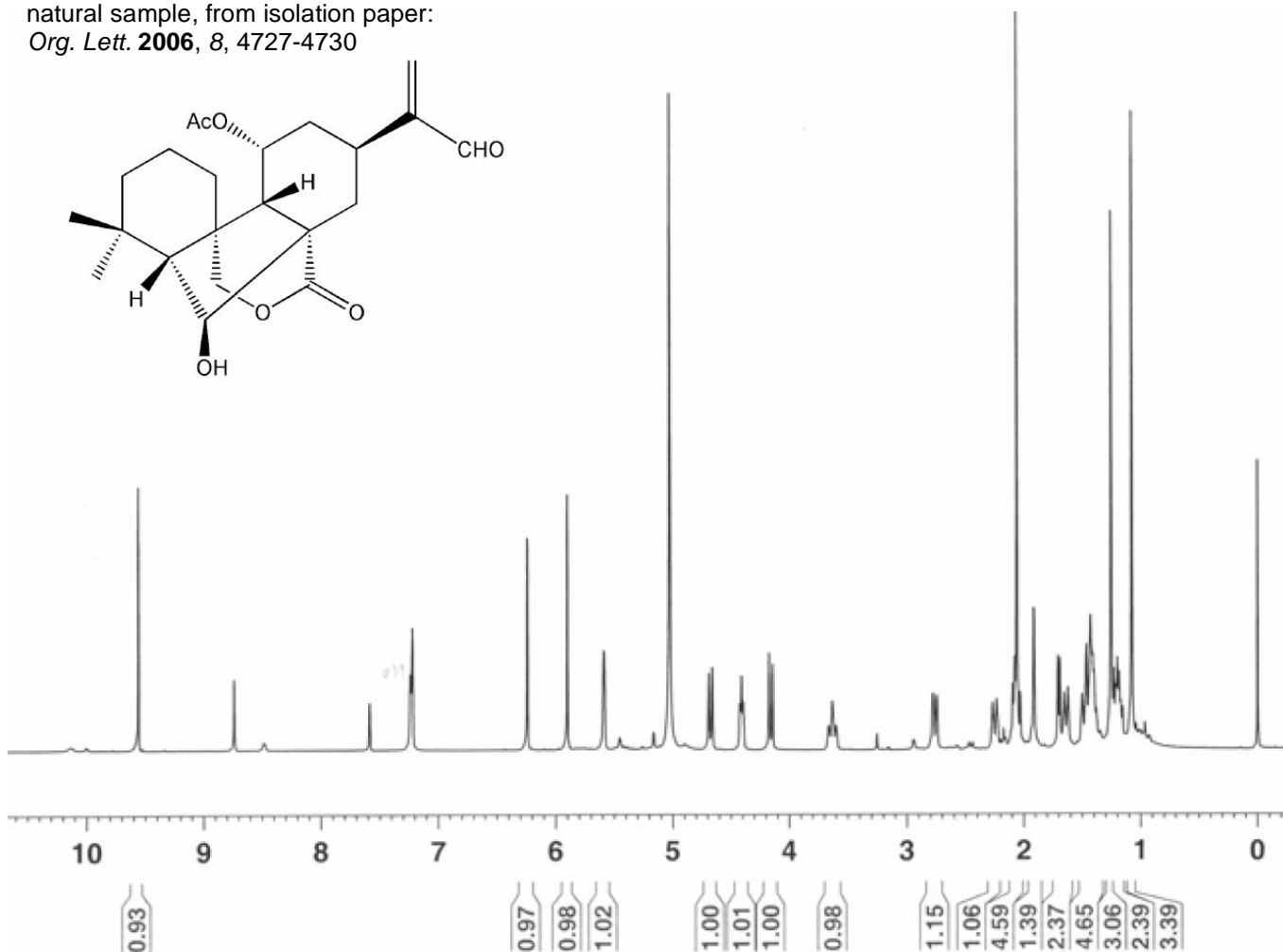
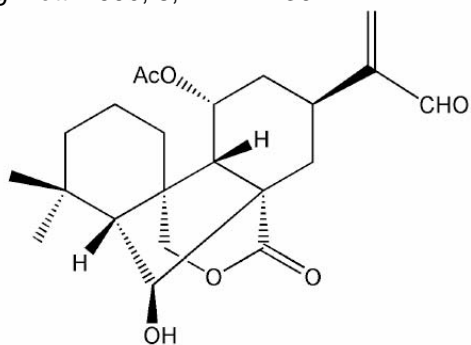
Line broadening 0.2 Hz

FT size 65536

Total time 23 min



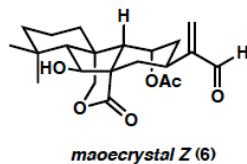
natural sample, from isolation paper:  
*Org. Lett.* **2006**, 8, 4727-4730



pie #40, operator: jcn

lax. delay 3.000 sec  
 lse 45.0 degrees  
 q. time 2.500 sec  
 dth 8000.0 Hz  
 8 repetitions  
 ERVE H1, 499.7217632 MHz  
 A PROCESSING  
 ne broadening 0.2 Hz  
 size 65536  
 al time 23 min

synthetic sample



maoecrystal Z (6)

